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INVESTIGATION OF FILM REINFORCED COMPOSITES FOR AIRCRAFT STRUCTURES

By

Earl E. Chadsey, Jr.

Frank Feakes

December 1969

U. S. ARMY AVIATION MATERIEL LABORATORIES
FORT EUSTIS, VIRGINIA

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NORTON RESEARCH CORPORATION
CAMBRIDGE, MASSACHUSETTS

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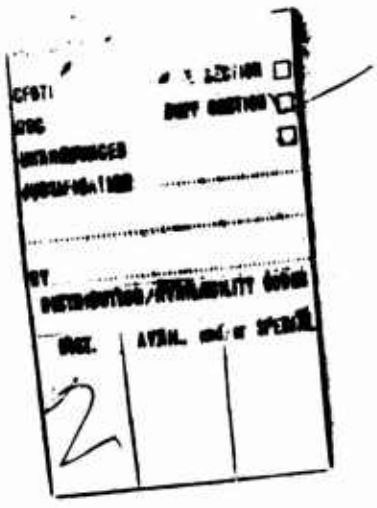

DEPARTMENT OF THE ARMY
HEADQUARTERS US ARMY AVIATION MATERIEL LABORATORIES
FORT EUSTIS, VIRGINIA 23604

This program was carried out under Contract DAAJ02-68-C-0091 with Norton Research Corporation, Cambridge, Massachusetts.

The data in this report are the result of research directed to the improvement of mechanical properties of boron thin-film reinforcement. Effort was made to reduce the type and number of flaws affecting strength of the reinforcement.

The report has been reviewed by the U.S. Army Aviation Materiel Laboratories and is considered to be technically sound. It is published for the exchange of information and the stimulation of future research.

Task 1F162204A17001
Contract DAAJO2-68-C-0091
USAAVLABS Technical Report 69-85
December 1969

INVESTIGATION OF FILM REINFORCED COMPOSITES FOR AIRCRAFT STRUCTURES

Final Report

By

Earl E. Chadsey, Jr.
Frank Feakes

Prepared by

Norton Research Corporation
Cambridge, Massachusetts

for

U.S. ARMY AVIATION MATERIEL LABORATORIES
FORT EUSTIS, VIRGINIA

SUMMARY

The main objective of this program was to develop improved mechanical properties of boron thin-film reinforcement. Optical examinations of laminates made by bonding together multiple layers of vacuum evaporated boron supported on polyimide substrates in earlier programs had shown significant variations in glue-line thickness and reinforcement planarity. Consequently, the present program emphasized methods of reducing these types of laminate imperfections. Three approaches were experimentally tested. The best results were obtained with laminates made with polyimide film (Kapton), 1/4 mil thick, as the substrate. This was vacuum coated on both sides with boron using a screen carrier in the vacuum coating operation. Multiple layers were bonded together to make laminates for mechanical testing. The optimum mechanical properties obtained were as follows: tensile strength, 56.6×10^3 psi; tensile modulus, 23.0×10^6 psi; flexural strength, 57.4×10^3 psi; flexural modulus, 23.1×10^6 psi; compressive strength, 90×10^3 psi; compressive modulus, 24.5×10^6 psi; shear modulus, 7.7×10^6 psi; and interlaminar shear strength, 13.8×10^3 psi. The density of typical laminates was 0.064 pci. While the laminates showed a high degree of isotropy in the plane of the reinforcement, variations in tensile strength associated with the direction of travel of the substrate through the coating chamber were observed. In addition, the test results showed relatively large deviations with the average values significantly lower than the optimum values reported above. To a large extent, the lower values could be attributed to variations in both the coating and laminating operations. A number of specific recommendations for eliminating strength-reducing factors resulted from the present work.

In general, the optimum results obtained for the boron-film composite in the present program show strength values which are comparable to other planar isotropic materials of importance to the aerospace industry such as aluminum alloys, titanium alloys, and pseudo-isotropic lay-up ($0^0 \pm 60^0$) of boron filament. On the other hand, the specific modulus of the boron-film composite is approximately 3 times that of both the aluminum and titanium alloys and approximately 2 times that of pseudo-isotropic boron-filament composites.

FOREWORD

The work reported herein was authorized by Contract DAAJ02-68-C-0091, Task 1F162204A17001, with the U.S. Army Aviation Materiel Laboratories, Fort Eustis, Virginia.

The authors acknowledge the contributions of the following people to the program: Professor A. S. Argon for continued stimulating advice and for his work with the fractography studies; Dr. G. E. Padawer for his contributions to the material studies and mechanical testing; also J. Webster, C. Bertolami, W. Rhinehardt, E. Gobbi, and L. Klein for their consistent efforts in film production, composite fabrication, and testing.

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D	total midspan deflection, in.
D_M	midspan deflection due only to bending moment, in.
D_S	midspan deflection due only to shear, in.
E	Young's modulus, psi
E_F	flexural modulus, psi
E_T	tensile modulus, psi
E^3	3-point bending modulus, psi
G_M	shear modulus of matrix, psi
G_{xy}	in-plane shear modulus, psi
G_{xz}	transverse shear modulus, psi
L	specimen span length, in.
P	applied load, lb
T	applied torque, in.-lb
t	specimen thickness, in.
V	vertical load through the specimen, lb
vf	reinforcement volume fraction
w	specimen width, in.
θ	angle of twist, radians
ν_{xy}	in-plane Poisson's ratio
σ_F	flexure strength, psi
τ	shear stress, psi

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INTRODUCTION

This report covers research work performed by Norton Research Corporation under Contract DAAJ02-68-C-0091 with the U. S. Army Aviation Materiel Laboratories, Fort Eustis, Virginia. The research program was directed toward optimization of the engineering properties of boron thin film reinforcement composites. Specific objectives were:

1. Preparation of boron thin film reinforcement materials by three concepts:

   a. Concept Number 1: Deposition of boron on thin polyimide resin films formed on a metallic base.

   b. Concept Number 2: Deposition of boron on 1/4-mil polyimide film bonded to a metallic base.

   c. Concept Number 3: Deposition of boron on 1/4-mil polyimide film.

2. Selection of the best of these three reinforcement materials by:

   a. Fabrication of test laminates from each material.

   b. Characterization of the performance of each test laminate by:

      (1) Determination of tensile stress-strain curves, tensile modulus, proportional limit, and ultimate strength.

      (2) Determination of the failure mechanism for each laminate by an optical inspection after the ultimate strength test.

      (3) Determination of the planarity of the reinforcement in each laminate by an optical inspection.

   c. Evaluation of the three candidate reinforcement materials by comparing the performance of the test laminates made from them.

3. Optimization of the process parameters to obtain the best reinforcement materials.

4. Fabrication of laminates using the best reinforcement

material (three of these laminates to be supplied to U. S. Army Aviation Materiel Laboratories, Fort Eustis, Virginia).

5. Testing of the remaining final laminates by Norton Research Corporation, to include the following determinations:

   a. Tensile stress-strain curves.

   b. Flexural modulus and ultimate strength.

   c. Shear modulus and interlaminar shear strength.

   d. Compression modulus and ultimate strength.

   e. Failure mechanism during ultimate strength tests.

6. Evaluation of the data obtained from the test program.

The work performed in preparing and evaluating reinforcement material for selection of the best concept is summarized in Section I. This work indicated that material made by deposition of boron on 1/4-mil polyimide film had the best mechanical properties when fabricated into a laminate. Consequently, material made by this concept was used to fabricate the final laminates. The fabrication and testing of the final laminates are summarized in Section II.

SECTION I: EVALUATION OF THREE CONCEPTS OF REINFORCEMENT PREPARATION

Concept Number 1 - Boron Reinforcement on Polyimide Resin Film Formed on a Metallic Base

The procedure for the fabrication of a composite based on the deposition of a boron reinforcing film on a thin polyimide substrate which had been formed on a metallic base included the following steps:

1. Coating of one side of 2-mil aluminum foil with a thin layer (0.1-0.2 mil) of polyimide resin (Monsanto Skybond 704).

2. Curing of the 704-polyimide film on the aluminum foil.

3. Coating of the 704-polyimide surface with a layer of boron by vacuum evaporation.

4. Fabrication of a primary laminate by bonding two pieces of the above material with the boron face to face.

5. Formation of a multilayered laminate by bonding a number of the primary 704-polyimide-boron units together with epoxy resins.

The procedure offered the possibility of coating substrates which were thinner than the thinnest polyimide film commercially available (1/4 mil). This in turn indicated a method of increasing the volume fraction of reinforcement in the final laminate. In addition, it was anticipated that the 2-mil aluminum would be sufficiently strong and stiff to produce a boron deposit which was flatter and less wrinkled than that obtained by coating an unsupported polyimide film.

The initial work was directed toward the determination of the time-temperature requirements for developing a high degree of cure and sufficient outgassing of the polyimide film. This was required to obtain satisfactory adhesion between the boron and polyimide in the subsequent boron coating operation. Table I shows the effect of different curing and outgassing conditions on coating adhesion. Adhesion improved as exposure time and temperature were increased. For all samples, there was no apparent thermal degradation of the 704-polyimide film. Samples 4 and 5 indicate that the vacuum oven produced a comparable outgassing effect in less time than the air oven. Sample 6 was exposed to curing and outgassing conditions comparable to those expected to prevail during continuous film

TABLE I. CURING AND OUTGASSING OF 704-POLYIMIDE FILMS FORMED ON ALUMINUM FOIL

Sample Number	Curing Stage Temperature (°C)	Curing Stage Time (min)	Oven Type	Outgassing Time at Temperature 107°C	124°C	163°C	185°C	Adhesion* of Boron Coating
1	225	45	None	-	-	-	-	Very poor
2	225	45	Vacuum	1 hr	-	-	-	Very poor
3	225	45	Vacuum	1 hr	17 hr	-	-	Poor
4	225	45	Vacuum	1 hr	17 hr	23 hr	-	Fair
5	225	45	Air	-	-	7 days	-	Fair
6	275	20	Vacuum	-	-	-	5 days	Good

*The degree of adhesion has been rated according to the fraction of the boron coating removed by the "Scotch Tape" test after exposure of the polyimide-boron material to boiling water for 2 hours. The ratings used were approximately as follows:

Percent Removed by Scotch Tape	Adhesion Rating
Greater than 20	Very poor
10-20	Poor
3-10	Fair
1-3	Good
0-1	Excellent

forming.

A small experimental coater was modified to permit thin polyimide films to be formed semicontinuously on 2-mil aluminum foil. The drive unit was designed to move material through the coater at speeds ranging from 0.02 to 0.6 foot per minute. This rate range permitted residence times in the oven section of the coater to be varied between 10 minutes and 300 minutes. The applicator unit was designed to form a 7-inch-wide film on a 9- to 12-inch-wide substrate. The 704-polyimide solution was transferred from a sump to the aluminum foil surface by a 7-inch-wide lamb's-wool paint roller. An idler roll behind the foil exerted pressure against the lamb's-wool roller to squeeze out excess solution.

Approximate conditions for semicontinuous operation of the experimental coater were determined by applying 704-polyimide solutions with a paintbrush to 6-inch-square areas of the moving foil. A 100-foot length of aluminum foil was then coated with a continuous, unbroken 704-polyimide film. The apparatus is shown in Figures 1, 2, and 3. A 7-inch-wide thin film of a 1:1 solution of Skybond 704-polyimide in 1-methyl, 2-pyrolidinone was applied to 10-inch-wide by 2-mil-thick aluminum foil by the lamb's-wool roller. The coated foil passed through the precuring stage and the curing oven, and the product was wound on a metal core. Temperatures in the 6-foot-long oven ranged from 270^{0}-310^{0}C, and foil transport speed ranged from 20 to 30 feet per hour. The roll of product was outgassed in a vacuum oven at a temperature of 185^{0}C for 5 days before it was installed in pilot-scale vacuum coating equipment for application of a boron reinforcement layer.

Two vacuum vapor deposition runs were made in which elemental boron was deposited on the aluminum supported polyimide film. In run 42-253-A1, operating conditions were defined. In run 42-253-A2, material was prepared for use in fabricating a laminate. Over 50 feet of boron-coated reinforcement material was produced in these two runs. Adherent boron coatings up to 0.08 mil thick were applied to polyimide films as thin as 0.06 mil. Heavier boron coatings were obtained, but the increased exposure times required led to some blistering of the 704-polyimide layer and poorer adhesion of the boron.

In order to develop satisfactory methods for the removal of the 2-mil aluminum, laminates were first made which did not contain boron. Two-ply laminates were fabricated from aluminum foil samples which had been coated with thin (0.045 to 0.065 mil) 704-polyimide films. The polyimide surfaces were

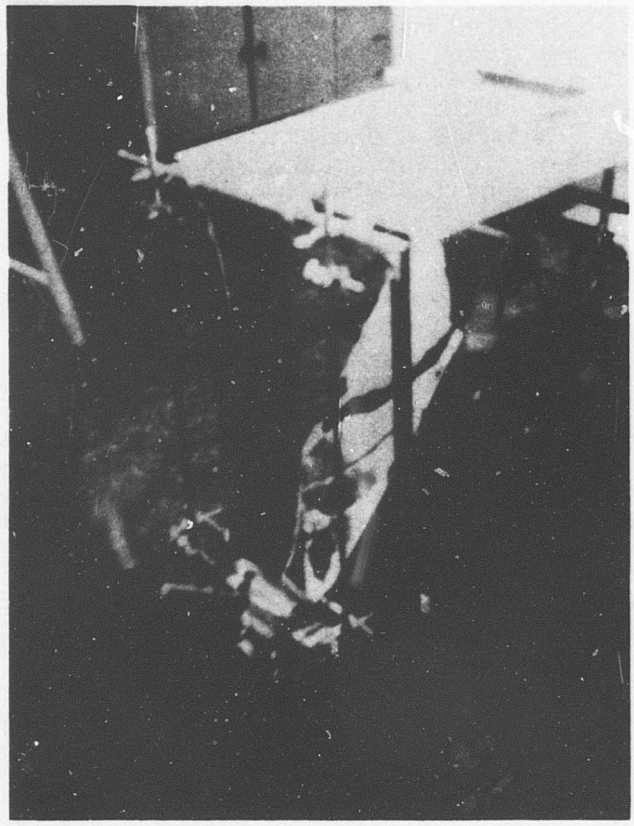


Figure 1. Experimental Plastics Coater, Input End, Showing Supply Roll, Applicator Unit, and Infrared Precuring Lamps.

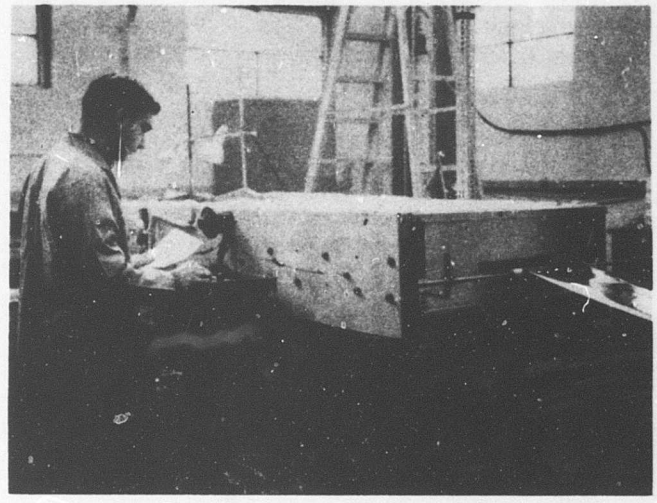


Figure 2. Experimental Plastics Coater, Oven Section, Showing Temperature Measurement Being Taken.

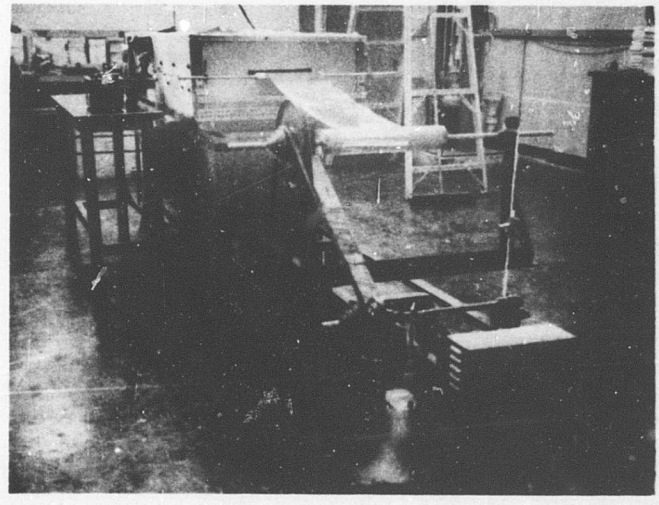


Figure 3. Experimental Plastics Coater, Output End, Showing Wind-up Roll and Drive Unit.

bonded together with epoxy (Union Carbide ERL-2256), and the laminates were cut into sample strips. These strips were then etched with various chemical solutions to determine the best solvent for removal of the aluminum covers. The results are shown in Table II. Dilute hydrochloric acid containing cupric sulfate removed the aluminum completely with no apparent degradation of the 704-polyimide film. The resulting polyimide/epoxy/polyimide sandwich was found to be strong enough to be handled without breaking. This suggested that the method was feasible for fabrication of boron-containing laminates.

TABLE II. CHEMICAL SOLUTIONS EVALUATED AS ETCHANTS FOR REMOVING ALUMINUM FROM AL/704-POLYIMIDE LAMINATES

Solution	Concentration (%)	Solution Time (minutes)	704 Condition
NaOH	10	30	Fair
	20	1.5	Good
	30	0.6	Poor
HCl	15	25	Fair
	25	2	Fair
	37	10	Good
HCL - 1% $CuSO_4$	15	4.5	Good
	25	2	Good
H_2SO_4*	15	No action	-
	25	No action	-
	35	No action	-

*With or without $CuSO_4$

Two secondary laminates, each containing 50 layers of boron, were fabricated from reinforcement material made in run 42-253-A2. In each case, the procedure used was as follows. Three 2-layer primary laminates were made by bonding pairs of 6-by-7-inch reinforcement sheets together with the aluminum on the outside. These primary laminates were then cut into 3/4-by-6-inch strips, and the outer aluminum layers were removed by chemical solution. The polyimide/boron/epoxy/boron/polyimide strips which were obtained by removing the aluminum covers were then fabricated into 50-ply secondary laminates by bonding 25 strips together. Both secondary laminates made in this

manner were tested "as pressed" without any trimming of the edges. The analysis of these results is given in the Discussion of Results Section.

Concept Number 2 - Boron Reinforcement on 1/4-mil Polyimide Bonded to a Metallic Base

The second procedure considered in this program was to bond commercially available polyimide film (1/4-mil DuPont Kapton) onto a metallic base before vacuum coating with boron. Again, the main goal was to provide mechanical support for the polyimide substrate in order to produce a flat, planar, unwrinkled reinforcement layer.

Two candidate metallic base materials were investigated: aluminum foil and aluminum wire screening. Aluminum foil was used in the initial experiments and demonstrated a basic problem with the method. Solvents and moisture in the polyimide resin used as the bonding agent remained trapped between the two relatively impervious layers during the curing period. This caused the polyimide film to wrinkle and blister on the foil base.

In order to provide an escape path for the volatile materials, aluminum wire screening was substituted for the aluminum foil. The screening was flattened in a press at a pressure of 1 ton per square inch before the bonding operation. Two samples were prepared in which the polyimide layers were flat and smooth enough for use as substrates in a vacuum coating operation. These samples were cured at 65°C for 1 hour and then at 150°C for 2 hours. They were then outgassed in a vacuum oven at 163°C for 5 days to remove volatile materials from both the polyimide bonding resin and the polyimide film. The outgassed samples were coated with boron carbide in an experimental batch coater which permitted more exact control of the individual samples. Each sample was mounted in the coater and preheated under vacuum conditions before application of the coating. The applied coatings, which were about 0.07 mil thick, were extensively cracked and, despite all the outgassing which the samples had received, adhesion of the coatings to the polyimide substrate was poor. While it is likely that additional and more rigorous outgassing would bring about some improvement in adhesion, this work was discontinued because of:

1. Difficulty of solvent removal when using a sheet metal base.

2. Uneven thickness of the layer of polyimide bonding

agent after removal of the aluminum screen. This was caused by the localized accumulation of resin at the regions of close contact between the 1/4-mil polyimide film and the wire in the screen.

Concept Number 3 - Boron Reinforcement on 1/4-mil Polyimide Film

During an earlier contract[1] this type of reinforcement material was prepared and tested in the form of fabricated laminates. The results indicated a potential for improvement. The main goal of this part of the present contract was to produce flatter material with less cracked reinforcement coatings. The work included both the preparation of reinforcement material and the development of improved laminating procedures.

The first efforts to reduce wrinkles and folds in the reinforcement material and cracks in the boron coating were directed toward refinement of previously existing procedures. Special care was taken in loading the polyimide film into the vacuum chamber for coating. The transport rolls were carefully aligned and tension controlled to give reduced stretching and distortion of the polyimide film. In addition, preheaters were installed to maintain a more uniform polyimide temperature during the vacuum coating operation. A flatter material resulted. One laminate was made to evaluate this material.

Most of the wrinkles in the reinforcement material were caused by expansion of the polyimide film when it was exposed to the unavoidable heat from the high temperature boron vapor source. Preheating of the film before coating reduced wrinkling due to uneven expansion but did not reduce the wrinkling attributed to an overall increase in the area of the film during preheating and coating. Other substrates were substituted for the aluminum foil carrier normally used to assist the polyimide film through the coating operation. Efforts were made to allow the polyimide film to expand uniformly in small areas rather than making major wrinkles and folds. Two batches of material were made for evaluation. Large random distortion was greatly reduced, and it appeared that flattening during laminating would produce none of the large wrinkles previously observed. The degree of adhesion between the boron and polyimide film was good. In all, 5 laminates were made to evaluate this type of material. The test results obtained with these laminates will be compared with those obtained under concept number 1 in Section I, Discussion of Results.

DEVELOPMENT OF LAMINATING PROCEDURES

Translation of the maximum potential of a reinforcement material into a composite demands laminating procedures which do not cause degradation or dilution of the desired mechanical properties. The procedure developed during the previous contract was modified to improve glue-line uniformity, to flatten individual laminate layers, and to reduce cracking of the boron coating.

Since it was possible that the higher pressures and elevated temperatures required for thinner glue-lines may cause greater cracking of the reinforcement, the laminating procedure was first modified by reduction of the laminating pressure. The lower pressures appeared to be effective in reducing reinforcement cracking but resulted in increased glue-line thickness and larger numbers of wrinkles.

One laminate was made without curing agent in the epoxy so that the sheets could be separated and inspected after simulation of the laminating procedure. This was done to assess the degree of reinforcement cracking during the laminate pressing operation. The laminate lay-up was precooled (0-5^{0}C) and cold-pressed in order to simulate the higher viscosity of the normally partly cured epoxy. Considerable cracking of the reinforcement layers occurred. The aspect ratio of the boron flake was reduced from about 1200 to 300. Nevertheless, separate theoretical studies at Norton Research Corporation have indicated that if the flake aspect ratio is in excess of about 100, high translation of the flake strength is possible in a laminate. Consequently, relatively high pressures were maintained for laminate fabrication. See Table III.

Another procedure which was adopted was to apply a thin precoat of adhesive to the boron surface of each sheet of reinforcement and then partially cure this before applying the main adhesive layer. This procedure appeared to have a beneficial effect in reducing the amount of reinforcement cracking.

A number of methods for producing a radial pressure distribution in the laminate during squeeze-out of the adhesive were investigated. The objective was to make the individual layers in the laminate more planar. The procedures tested included control of the viscosity of the adhesive by the temperature-pressure sequence and inclusion of layers of pressure distributing materials in the laminate pressing assembly. The temperature-pressure sequence was programmed to permit application of the laminating pressure at the moment when the glue was viscous enough to exert a radial viscous force as it flowed outward from between the layers of reinforcement. This

TABLE III. DETAILS OF FABRICATION CONDITIONS FOR EVALUATION LAMINATES

Specimen Number	Size (w x l) (inches)	Number of Layers	Precoat Conc*	Precoat Precure (min at 225°F)	Adhesion Coat Conc*	Adhesion Coat Precure (min at 225°F)	Applied Pressure (psi)	Cure	Post-Cure
BORON 704-POLYIMIDE FILM (CONCEPT 1)									
88-122 Primaries	6 x 7	2	1:15**	40	1:1**	23.5	500	225°F/1 hr	300°F/1hr
Secondary	3/4 x 4	25	None	-	1:1**	22	500	"	"
88-146 Primaries	6 x 7	2	1:30**	None***	1:1**	20	500	"	"
Secondary	3/4 x 4	25	1:30**	None***	1:1**	20	500	"	"
BORON 1/4-MIL POLYIMIDE FILM (CONCEPT 3)									
88-12	5 x 5	19	None	-	1:1**	27	500	225°F/1 hr	300°F/1hr
88-18	5 x 5	19	1:15**	45	1:1**	27	500	"	"
88-48	3/4 x 5	15	1:15	45	1:1	27	500	"	"
88-54	3/4 x 5	30	1:15	40	1:1	22	750	"	"
88-74	3/4 x 5	39	1:15**	40	1:1**	22	750	"	"
11-116	3/4 x 5	40	1:15**	40	1:1**	22	750	"	"

*Concentration

**With 0.4 part of silane per 100 parts by weight of 1:0 epoxy

***No precure - 1:30 rinse followed directly by 1:1 adhesive solution

technique was found to be moderately effective but difficult to control. Partially confined layers of silicone rubber or untreated polyimide film were found to be more effective in varying degrees in equalizing pressure across the entire laminate surface. Flatter, less lens-shaped laminates were obtained with this technique.

Fabrication of laminates in two stages also showed promise for reduction of the carry-over of reinforcement wrinkles into the laminate. One 2-layer laminate and one 4-layer laminate were made from material which was quite wrinkled. The 4-layer laminate or tape contained a few wrinkles, but it was a considerable improvement over the material used to make it. The 2-layer laminate contained no wrinkles and was quite flat and smooth. This technique is a promising one, requiring further development.

MECHANICAL TESTING OF EVALUATION LAMINATES

Laminates were sectioned into test coupons and fitted with grips and strain gages, as previously described.[1] Tensile tests for specimens A (parallel to the film transport direction) and for B and C (transverse to the film transport direction) also followed the methods described in Reference 1.

Uniaxial tensile tests were employed to evaluate the relative merits of the candidate preparation concepts. Test results are shown in Table IV.

DISCUSSION OF RESULTS

Of the three concepts considered in this part of the program, concept number 2, in which 1/4-mil polyimide film was bonded to a metallic base, appeared to be the least promising. The basic requirements of a well outgassed substrate for the vacuum vapor deposition step and a low volume fraction of adhesive in the final composite were not met. Both the polyimide film and the aluminum foil investigated as the base materials have such low permeabilities that solvents in the adhesive could not be adequately removed. This caused the film to wrinkle and bubble on the base. Substitution of a flattened wire screen as a base material reduced the outgassing problem. However, adhesive flowed into the meshes when the film was attached to the screen and remained as mounds when the screen base was removed by chemical solution. It was apparent that these mounds would increase the volume fraction of adhesive in a laminate. The unevenness would also decrease the planarity of the layers in the laminate. Therefore, concept number 2 was abandoned.

TABLE IV. SUMMARY OF TENSILE PROPERTIES OF EVALUATION LAMINATES

Spec No.	Density (pci)	Boron Content (Vol %)	Elastic Modulus (10^6 psi)	PL Stress (10^3 psi)	Max Stress (10^3 psi)	Max Stress / VFB*	PL Strain (%)	E / VFB* (10^6 psi)
BORON 704-POLYIMIDE FILM (CONCEPT 1)								
88-122	0.0486	16.7	8.2	7.9	10.7	64	0.097	49
88-146	0.0531	28.6	11.7	14.4	30.7	107	0.123	41
BORON 1/4-MIL POLYIMIDE FILM (CONCEPT 3)								
88-12A**	0.0658	46.1	23.7	16.2	31.4	68	0.070	51
B***	0.0642	46.1	20.4	10.4	24.1	52	0.081	44
88-18A**	0.0598	35.2	19.3	18.9	29.9	85	0.098	55
B***	0.0596	35.2	18.8	11.7	22.8	65	0.062	53
88-48	0.0555	27.5	15.1	15.3	29.4	107	0.101	55
88-54	0.0634	38.4	19.7	21.6	45.0	117	0.110	51
88-74	0.0642	44.1	23.0	23.1	56.6	128	0.101	52
88-116	0.0624	40.7	22.4	24.4	36.5	90	0.109	55

*Volume Fraction B

**Sample A stressed parallel to film transport direction

***Sample B stressed perpendicular to film transport direction

Laminates were fabricated from material made by the other two concepts. Their properties are shown in Tables III and IV. The boron content ranged from 16.7 to 46.1 volume percent. The highest volume fractions were obtained with material containing 1/4-mil polyimide film. Higher volume fractions of boron were not obtained in the concept number 1 laminates for two reasons. First, in laminate number 88-122 the primary glue-line was excessive. Second, attempts to increase the boron thickness on the 704-polyimide resulted in thermal degradation of the 704-polyimide. While the range of coating parameters was varied in the present program, it was evident that conditions had not been optimized. It is not unlikely that a different combination of coating rate and film transport speed would permit the deposition of thicker boron films without excessive thermal degradation.

The tensile modulus values were higher for the material on the 1/4-mil polyimide film. Comparison of laminates 88-48 and 88-146, which contained about the same concentration of reinforcement, indicated a better translation of the modulus into the composite for the film-based material.

The highest ultimate tensile strength obtained was 56.6×10^3 psi for the 1/4-mil film laminate 88-74. Other values obtained for this concept were as high or higher than the high value of 30.7×10^3 psi obtained for the 704-polyimide material.

The proportional limit strain is considered to indicate the strain at which the reinforcement begins to fail (see Table III of Reference 1). Comparing the performance of concept number 1 and concept number 3 materials, it is apparent that the last four of the concept number 3 samples (88-48,54,74, 116), as well as the concept number 1 samples (88-122, 146), exhibited proportional limit strains of 0.1 percent or somewhat greater (the highest value was 0.123 percent for number 88-146). The maximum failure stresses, however, were exhibited by the concept number 3 materials.

In general, concept number 3 yielded composites with the higher properties. This pertained to volume fraction of reinforcement, tensile modulus, proportional limit stress, and ultimate strength. These results indicated that concept number 3 should be used to prepare materials for the final laminates. Preparation of material on 704-polyimide film formed on a metallic base, however, remained an interesting and promising concept even though the strength of the best laminate made by it had about half the strength of the best laminate made with 0.25-mil polyimide material. The method of concept number 1 has the possibility of yielding composites with reinforcement concentrations greater than 50 percent. Further

development would be needed to determine the thinnest 704-polyimide film which can be coated with boron and subsequently handled as a 2-ply/polyimide laminate. In addition, it would be necessary to develop procedures for increasing the thickness of the boron coating without blistering the thin 704-polyimide layer.

SECTION II: FABRICATION AND TESTING OF FINAL LAMINATES

Boron was vacuum deposited on 1/4-mil polyimide film using the technique described in Section I of this report. In general, a thinner coat (approximately 0.10 mil) was deposited on one side of the substrate and a thicker coat (approximately 0.15 mil) on the other.

The coated material was inspected, and samples of the best material were taken for tests and measurements. The thickness and the adherence of the boron coating were ascertained. Adhesion samples were boiled in water for 2 hours and then were subjected to a "Scotch Tape" test. Material which showed good adhesion after the 2-hour water boil was used for making laminates. (See Table III for method of rating the degree of adhesion.)

Selected material was cut to size for laminate lay-up. All final laminates were 5 inches by 4 inches. Individual sheets were coated with a thin protective layer of adhesive which was partly cured to make it dry to the touch. The sheets were then cut to laminate size for lay-up.

Five final laminates were made. Three of these, each 40 layers thick, were laid up with the film transport direction parallel for all sheets. Two were made so that the film transport direction was at right angles on alternate sheets. These were called cross-ply laminates. One cross-ply contained 40 layers; the other contained 36 layers. The resin system used was Union Carbide ERL-2256 together with Curing Agent Z. Vacuum laminating techniques were applied as outlined in the following procedure:

1. Material to be laminated was sampled, cut to size (approximately 7 inches by 5 inches), weighed, and measured.

2. Sheets were cleaned by light brushing on both sides in warm solvent baths followed by a thorough spray rinse with clean room-temperature solvent. Three solvents, triclene, acetone, and alcohol, were used in sequence.

3. The sheets were air-dried for 10 minutes and then wet spray-coated with an adhesive mixture of ERL-2256 (100 parts by weight), curing agent Z (20 parts by weight), and silane (0.4 part by weight), diluted with 15 times its volume of methyl ethyl ketone.

4. The sheets were then drained in air for 5 minutes and hung in an air oven at 225°F for 23 minutes.

5. The sheets were then laid up with a 1-inch-diameter button of undiluted vacuum outgassed adhesive mixture between each layer. Each button contained about 1 cubic centimeter of resin.

6. A pressure distributing section was included in the laminate package. This consists of two silicone rubber sheets 4 inches by 5 inches by 1/8 inch separated by a stainless steel sheet 5 mils thick.

7. The assembled laminate package was evacuated at room temperature to 200 microns without applying any pressure to the laminate.

8. Heat was applied to bring the laminate to a temperature of 225°F. This temperature was held for 5 minutes with no pressure on the laminate.

9. Pressure was applied to the package and brought up to 750 psi over a 2-minute period.

10. Cure conditions were held at 225°F and 750 psi for 1 hour.

11. The temperature was raised to 300°F.

12. Post-cure conditions were held at 300°F and 750 psi for 1 hour.

After completion of the post-cure, the press was water-cooled and the laminate was removed and cut to size. Accurate size and weight measurements were then made to calculate average density and glue-line thickness. The laminates were then tested. The fabrication details of the final laminates are shown in Table V.

MECHANICAL TESTING OF FINAL LAMINATES

Laminates number 121-72 (40 plies, parallel lay-up) and number 121-84 (40 plies, oriented 0° and 90° in alternation) were tested for tensile and compressive strength and stiffness, as well as for torsional stiffness and interlaminar shear. The 4-by-5-inch laminate plates were sectioned according to the cutting schedule, Figure 4.

Tensile specimens A and B were tested as described previously.[1]

TABLE V. FABRICATION DETAILS OF FINAL LAMINATES

Specimen Number	Number of Layers	Precoat Conc*	Precoat Precure (min at 225°F)	Adhesive Bead Conc*	Adhesive Bead Precure (min at 225°F)	Lay-Up Orientation	Applied Pressure (psi)	Cure	Post-Cure
121-67	40	1:25	23	1:0	None	Parallel ply	600	225°F/1 hr	300°F/1 hr
121-72	40	1:25	23	1:0	None	Parallel ply	750	225°F/1 hr	300°F/1 hr
121-76	40	1:25	23	1:0	None	Parallel ply	750	225°F/1 hr	300°F/1 hr
121-80	36	1:25	23	1:0	None	Cross ply	750	225°F/1 hr	300°F/1 hr
121-84	40	1:25	23	1:0	None	Cross ply	750	225°F/1 hr	300°F/1 hr

*Concentration

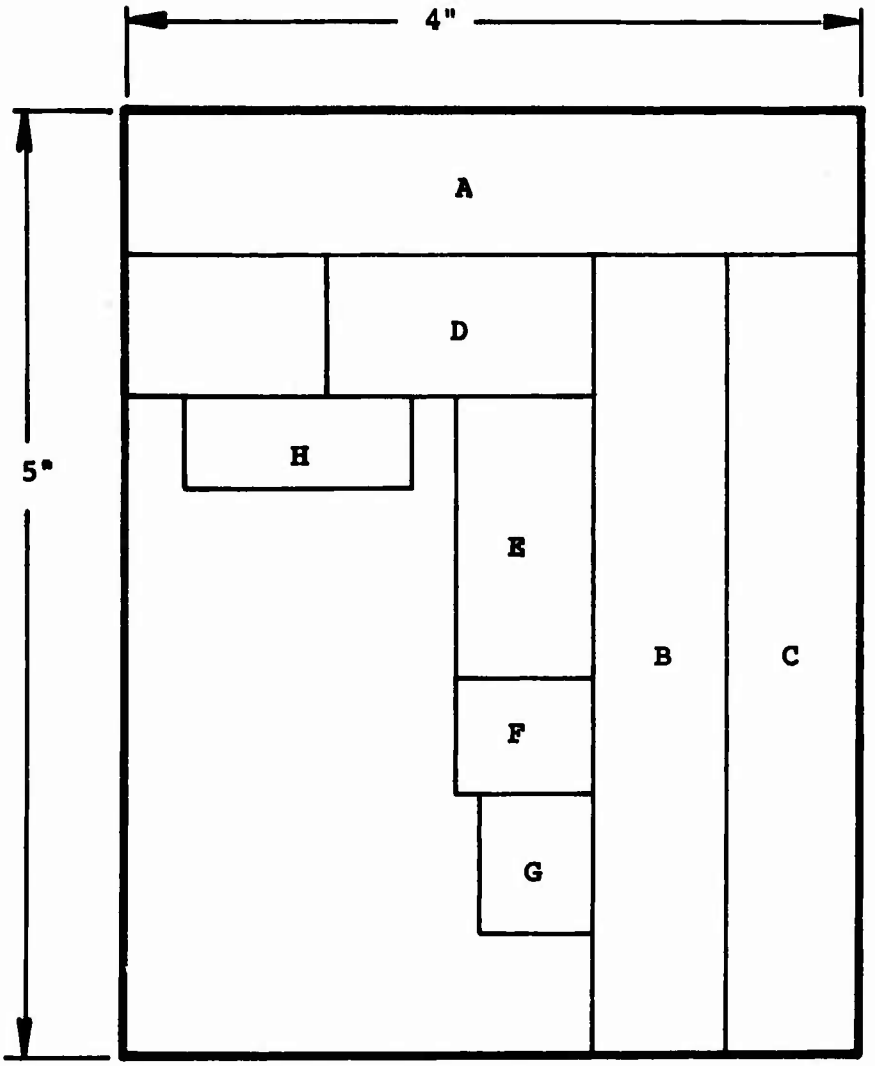


Figure 4. Laminate Cutting Schedule.
A,B - Tensile test
C - Torsional modulus (nondestructive), flexural test
D,E - Compression test, long specimen
F,G - Compression test, short specimen
H - Shear strength test

The in-plane shear modulus G_{xy} of specimen C was tested non-destructively in a simple torque-twist apparatus, shown in Figure 5. The in-plane shear modulus G_{xy} was computed by

$$G_{xy} = \frac{3TL}{wt^3\theta} \quad \text{(Ref. 2)} \qquad (1)$$

where T = applied torque (in.-lb)
L = specimen length between slotted grips (in.)
w = specimen width (in.)
t = specimen thickness (in.)
θ = angle of twist (rad)

The uniaxial compressive behavior was tested by two methods, illustrated in Figures 6a and b. Figure 6a shows the short specimen test, in which a specimen plate about 0.6 inch high and 0.75 inch wide was compressed between hardened steel platens. The specimen ends were fixed against rotation by 0.25-inch steel wedges, leaving about 0.1 inch of specimen height free and unsupported. Strain gages were mounted on both faces to generate stress-strain curves. The cross-head displacement rate was 0.02 inch per minute.

In an alternate setup, the overall specimen height was 1.5 inches, leaving 1 inch free height between the end-restraining wedges. To prevent Euler buckling in this region, lateral supporting fingers were clamped to the specimen, as shown in Figure 6b. The buckle wavelength developed at the failure point was thus limited effectively to the finger spacing (0.010 inch), as discussed further below.

A transverse shear loading jig was used to measure the interlaminar shear strength. As shown in Figure 7, the applied tensile pull imposes a transverse force on the specimen which is uniform, end to end, and which was assumed to have a parabolic distribution, top to bottom of the specimen. (This implies that the maximum longitudinal shear stress τ occurs at the center with magnitude 3/2 times the average.) Other normal and shear stresses due to the bending moment in the specimen are superimposed on the longitudinal shear stresses, but are ignored here. The test results therefore represent good lower bounds.

The actual interlaminar shear strength of the laminates is at least as high as, or higher than, the values given. The shear stress τ was computed by

$$\tau = \frac{3V}{2tw} \qquad (2)$$

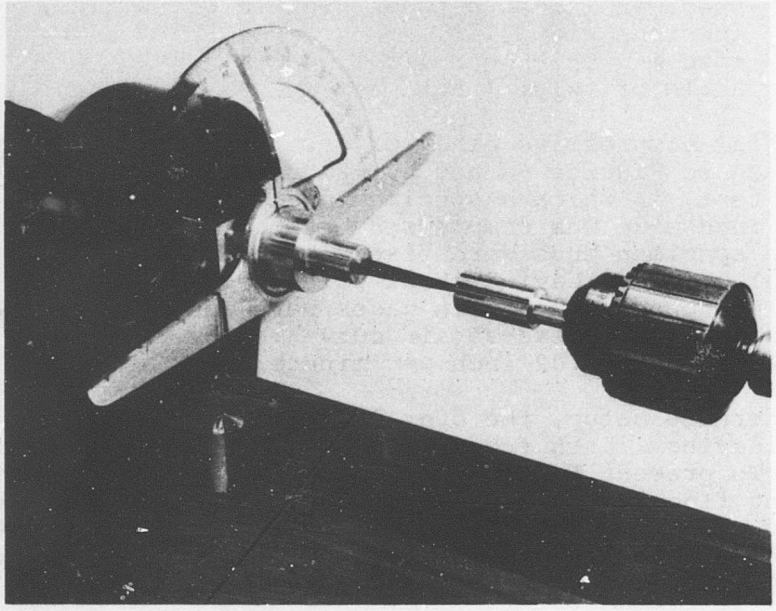
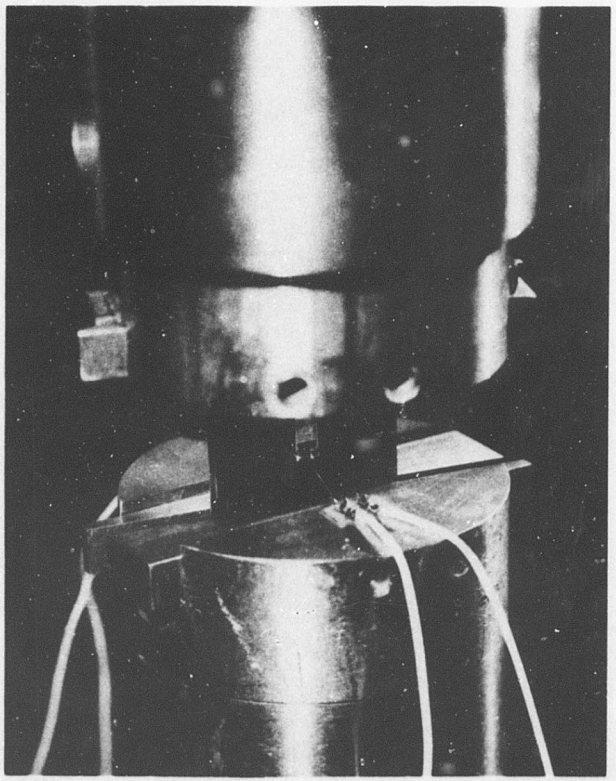
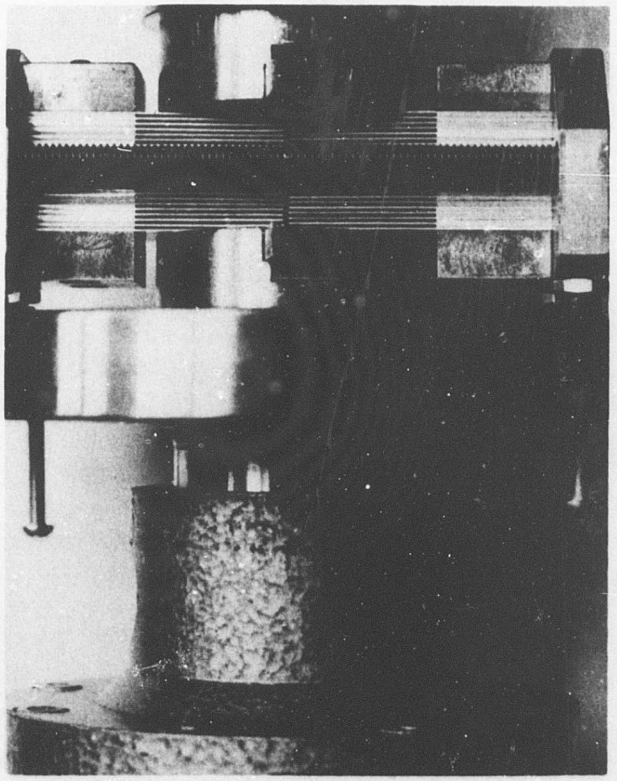


Figure 5. Shear Modulus Apparatus.

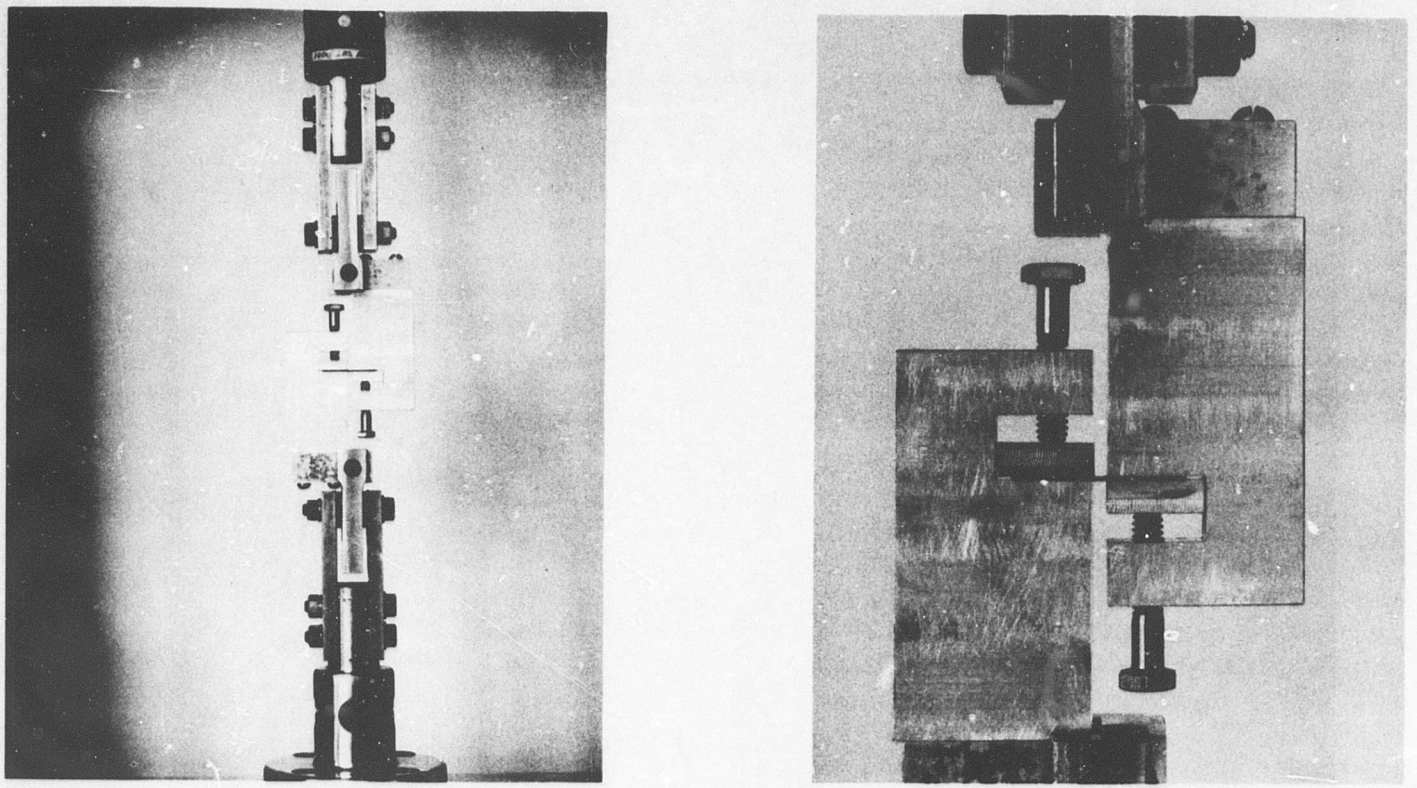


a. Short Specimen Test Jig.



b. Finger Support Apparatus.

Figure 6. Uniaxial Compression Test Assemblies.



a. Overall View.

b. Close-up View.

Figure 7. Interlaminar Shear Jig.

where V = vertical load through the specimen (lb)
t = specimen thickness (in.)
w = specimen width (in.)

The flexure test (specimen C) included plate flexure by 3-point bending over a 2.25-inch span to measure the bending modulus, followed by loading to destruction by 3-point bending over 0.75-inch "beam" span to measure the ultimate flexural strength. The 3-point bending modulus E3 was computed by

$$E3 = \frac{PL^3}{4wt^3D} \tag{3}$$

where P = applied load (lb)
D = corresponding total midspan deflection (in.)
L = specimen span length (2.25 in.)
w = specimen width (in.)
t = specimen thickness (in.)

The flexure strength σ_F was computed by

$$\sigma_F = \frac{3PL}{2wt^2} \tag{4}$$

where P = maximum midspan load (lb)
L = specimen span length (0.75 in.)
w = specimen width (in.)
t = specimen thickness (in.)

The load strain curves have been reproduced as stress-strain curves in Figures 8 through 12. In the case of the laterally supported compression specimens (121-72D and E, 121-84F and G), where strain gages could not be used, the Instron load/crosshead displacement curves were converted to stress-strain curves by accounting for the calibrated machine stiffness.

DISCUSSION OF RESULTS

In general, the mechanical characteristics of these laminates were quantitatively of greater magnitude than those reported in earlier work (see Reference 1). Tensile moduli ranged from 16.15 to 21.6 x 10^6 psi, and ultimate tensile strengths ranged from 23.9 to 36.5 x 10^3 psi. In addition, compressive strengths from 41.4 to 90.3 x 10^3 psi (average = 72 x 10^3 psi) were measured. See Tables VI and VII.

The interlaminar shear strengths, from 11.92 to 13.85 x 10^3 psi, indicated satisfactory adhesion at the boron/polyimide and at the boron/epoxy resin interfaces.

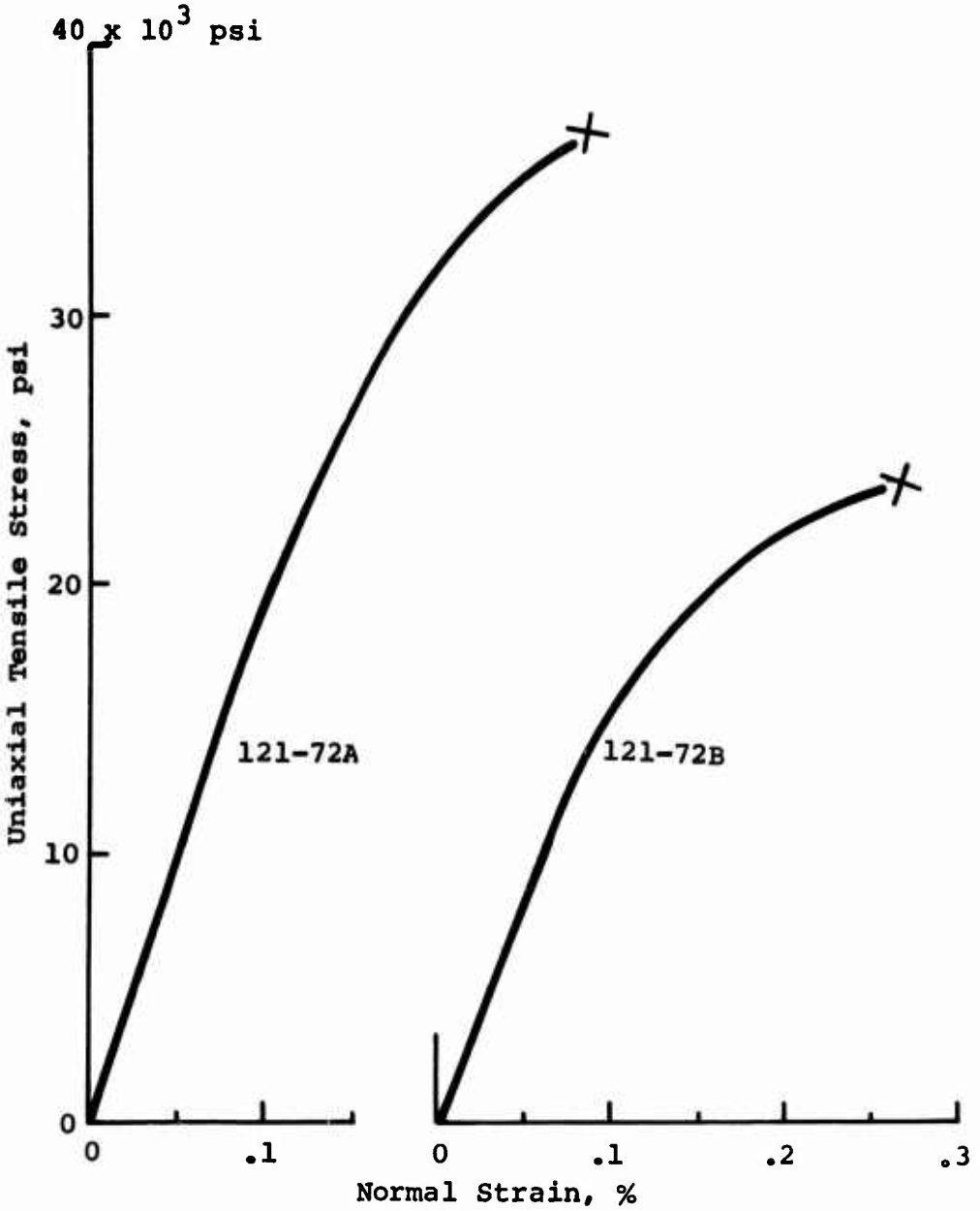


Figure 8. Stress-Strain Curve, Specimens 121-72A and 121-72B.

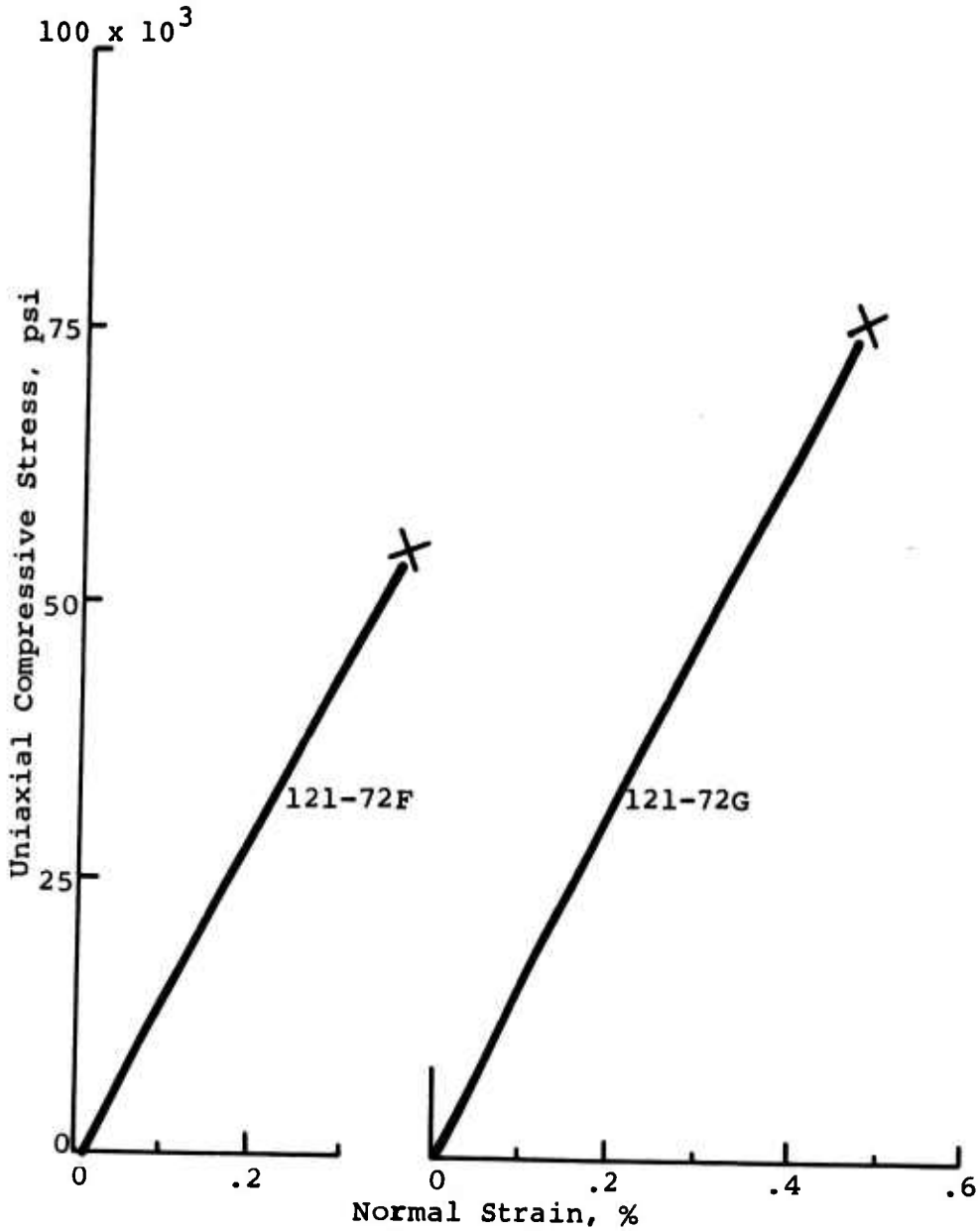


Figure 9. Stress-Strain Curve, Specimens 121-72F and 121-72G.

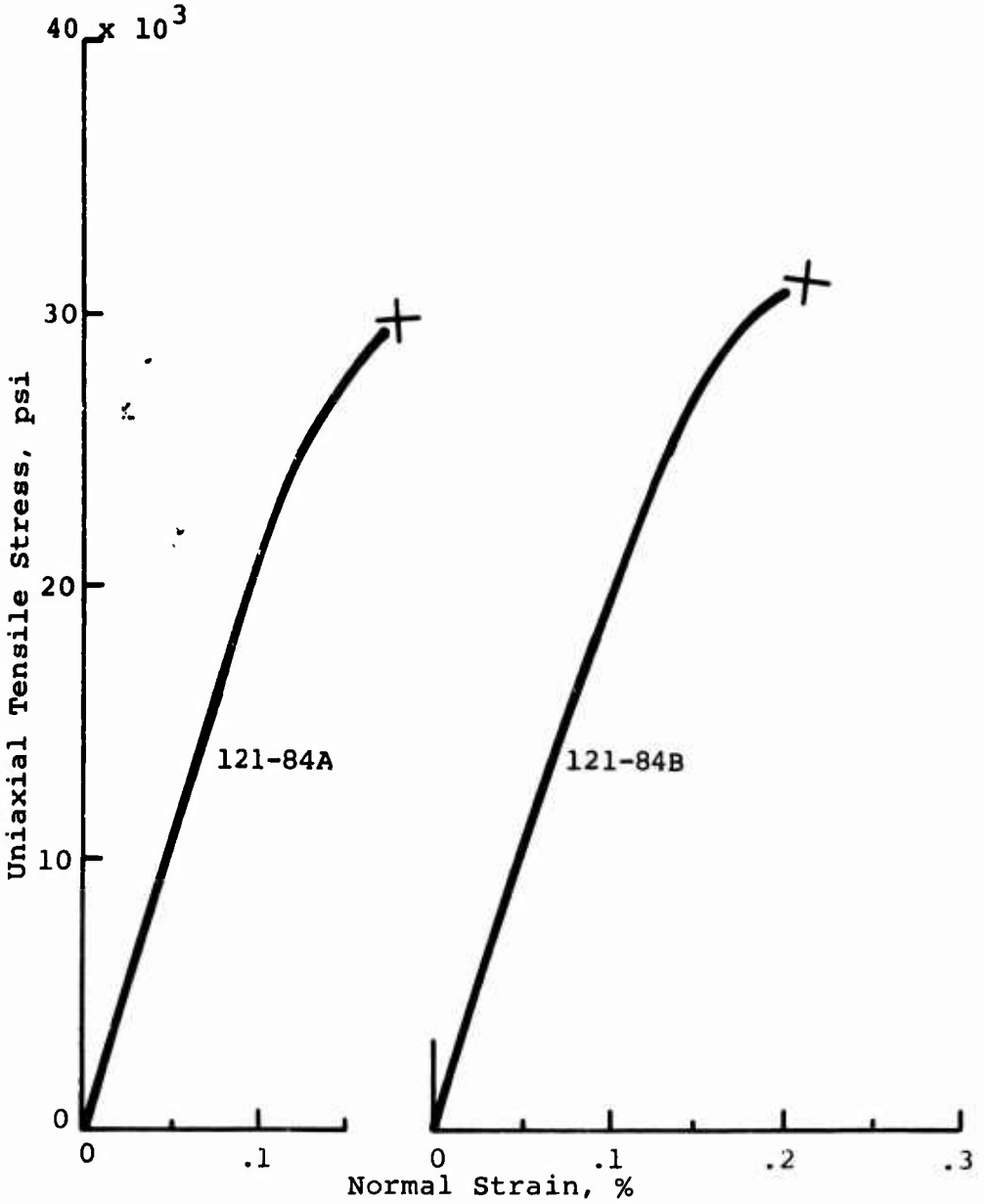


Figure 10. Stress-Strain Curve, Specimens 121-84A and 121-84B.

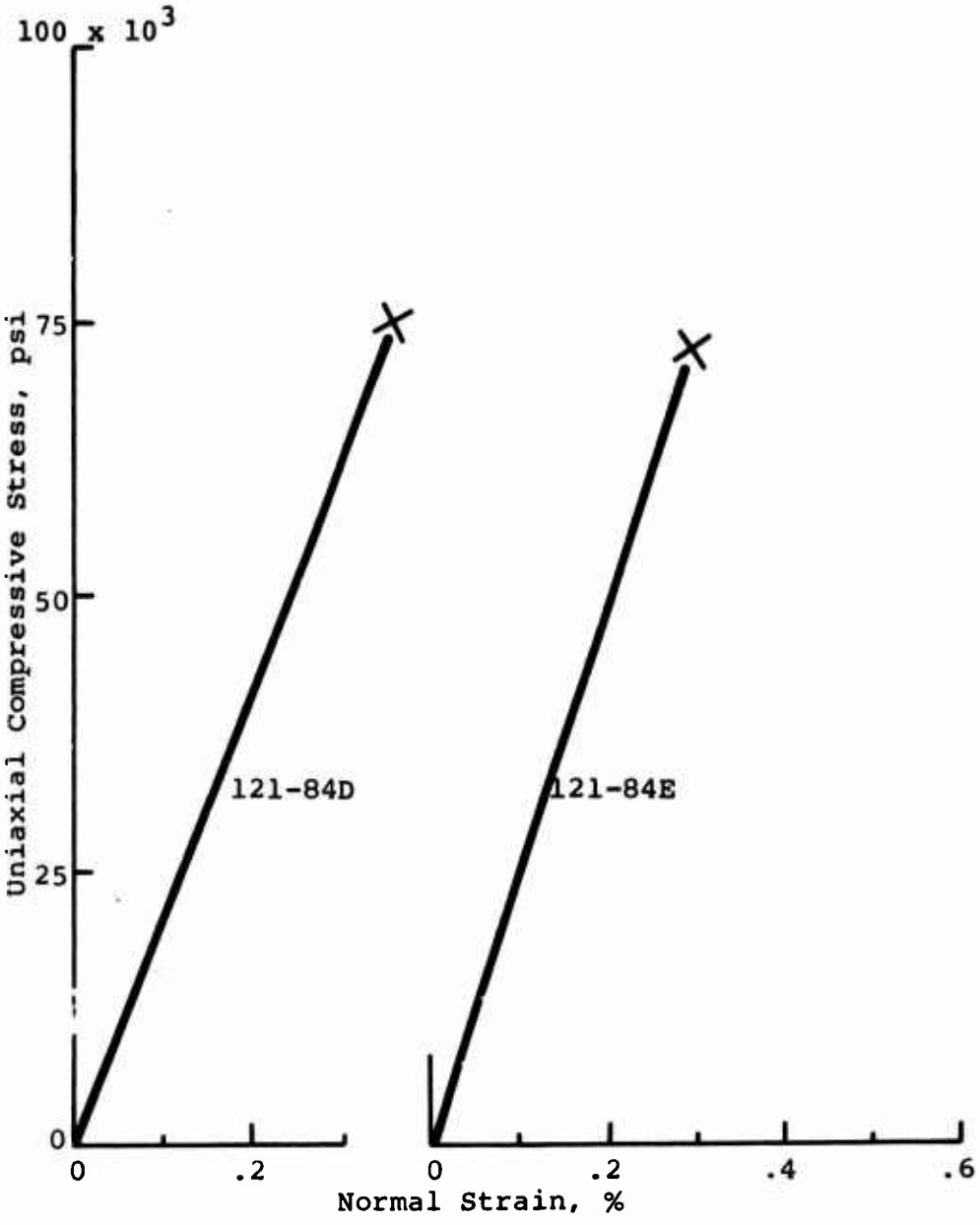


Figure 11. Stress-Strain Curve, Specimens 121-84D and 121-84E.

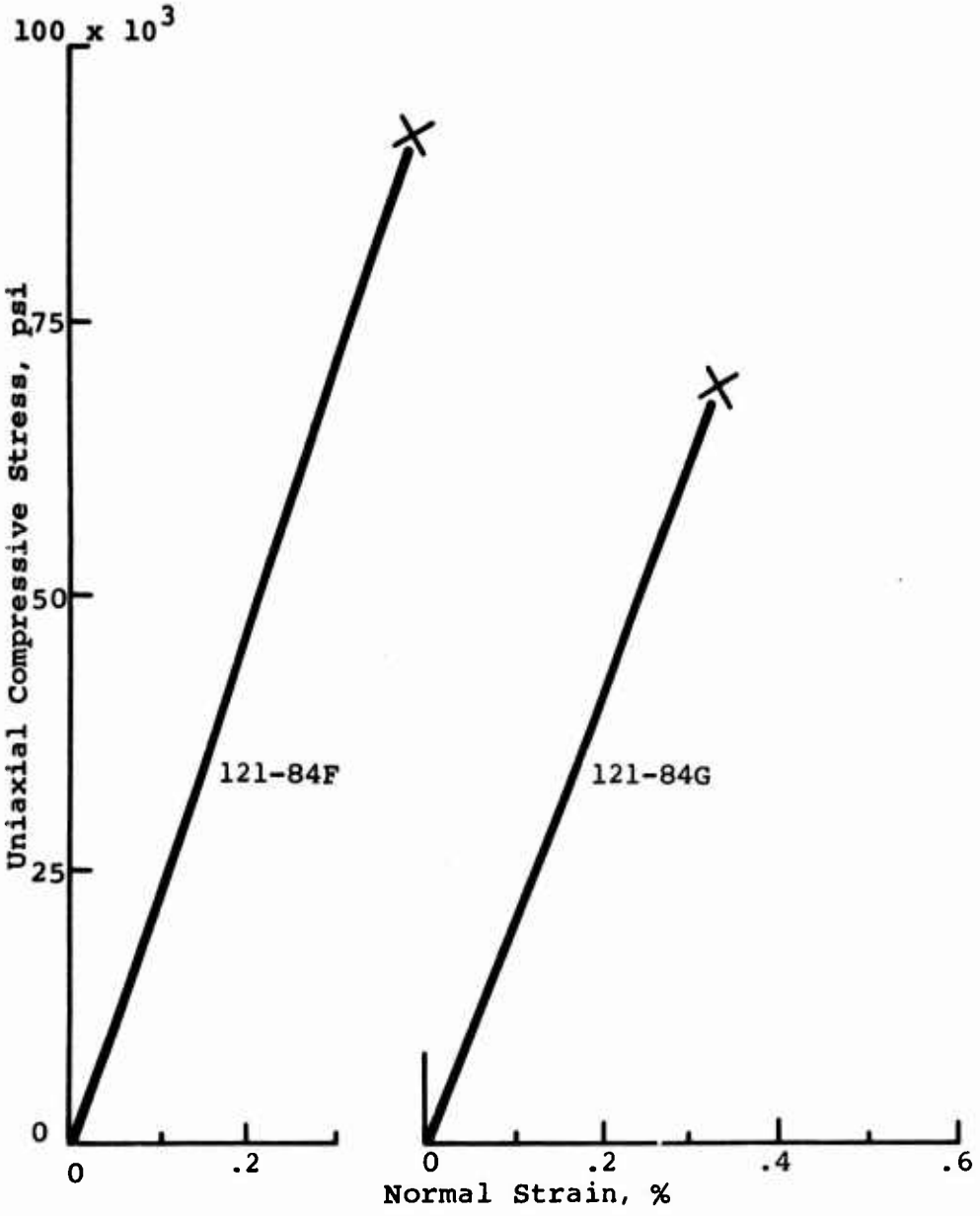


Figure 12. Stress-Strain Curve, Specimens 121-84F and 121-84G.

TABLE VI. MECHANICAL PROPERTIES: COMPOSITE NO. 121-72; VOL % BORON 39.2; SUBSTRATE - 1/4-MIL POLYIMIDE FILM

Spec No.	Density (pci)	Test Method	Elastic Modulus (10^6 psi)	PL[g] Stress (10^3 psi)	Max Stress (10^3 psi)	Failure Strain (%)	Remarks
A	.063	T[a]	19.4	21.4	36.5	.29	// to transport direction
B	.062	T[a]	16.2	9.3	23.9	.26	⊥ to transport direction
C	-	E3[b]	15.4	-	-	-	2.25-inch span
C	-	F[c]	13.7	-	49.5	-	0.75-inch span
C	-	F[c]	15.7	-	55.8	-	0.75-inch span
C	-	F[c]	10.0	-	39.9	-	0.75-inch span
C	-	G[d]	7.7	-	-	-	⊥ to transport direction
D	-	C[e]	-	-	78.8	-	// to transport direction
E	-	C[e]	-	-	41.4	-	⊥ 1.0-inch free length, ends fixed
F	-	C[e]	14.9	-	54.8	-	// to transport direction
G	-	C[e]	15.9	-	75.5	-	⊥ 0.1-inch free length, ends fixed
H	-	IS[f]	-	-	13.8	-	// to transport direction
H	-	IS[f]	-	-	11.92	-	⊥ to transport direction

(a) uniaxial tensile test
(b) three-point bending modulus
(c) tensile strength in flexure
(d) strip torsional test
(e) compression, fixed-ended column
(f) interlaminar shear test
(g) proportional limit

TABLE VII. MECHANICAL PROPERTIES: COMPOSITE NO. 121-84; VOL % BORON 44.7; SUBSTRATE - 1/4-MIL POLYIMIDE FILM

Spec No.	Density (pci)	Test Method	Elastic Modulus (10^6 psi)	PL[g] Stress (10^3 psi)	Max Stress (10^3 psi)	Failure Strain (%)	Remarks
A	.063	T[a]	21.6	19.0	29.4	.18	40 layer cross-ply
B	.065	T[a]	21.0	15.5	30.5	.21	-
C	-	E3[b]	22.1	-	-	-	2.25-inch span
C	-	F[c]	15.4	-	56.3	-	0.75-inch span
C	-	F[c]	15.4	-	56.5	-	0.75-inch span
C	-	F[c]	15.1	-	57.4	-	0.75-inch span
C	-	G[d]	6.9	-	-	-	-
D	-	C[e]	20.4	-	74.8	-	0.1-inch free length, ends fixed
E	-	C[e]	24.5	-	72.3	-	0.1-inch free length, ends fixed
F	-	C[e]	-	-	90.3	-	1.0-inch free length, ends fixed
G	-	C[e]	-	-	68.2	-	1.0-inch free length, ends fixed
H	-	IS[f]	-	-	13.6	-	.020-inch gage length

(a) uniaxial tensile test
(b) three-point bending modulus
(c) tensile strength in flexure
(d) strip torsional test
(e) compression, fixed-ended column
(f) interlaminar shear test
(g) proportional limit

The measured values of in-plane shear modulus G_{xy} from 6.99 to 7.66 x 10^6 psi demonstrated the effectiveness of the planar reinforcement, in contrast to the well-known weakness of filamentary composites, in this respect. The observed shear modulus values may be related to the corresponding tensile modulus values E very nearly by

$$G_{xy} = \frac{E}{2(1 + \nu_{xy})} \tag{5}$$

where ν_{xy} = in-plane Poisson's ratio, measured at about .1 to .2 in Reference 1.

This relationship holds strictly only in the case of isotropic, homogeneous, linearly elastic solids, and its applicability in the present case emphasizes the quasi-isotropic behavior of planar-reinforced composites.

The flexural moduli E_F observed in specimen C (test method F) were lower than the corresponding tensile moduli E_T. This may be explained by the short span of 0.75 inch which was employed for this test. In 3-point bending, the midspan deflection D_M due only to the bending moment is

$$D_M = \frac{PL^3}{4wt^3E3} \tag{6}$$

where the symbols have been defined in equation 4.

The additional midspan deflection D_S due only to shear is

$$D_S = \frac{3PL}{10wtG_{xz}} \quad \text{(Ref. 2)} \tag{7}$$

where G_{xz} = effective transverse shear modulus of the laminar composite (this is not the same as G_{xy} of eq. 1).

For purposes of this discussion, G_{xz} may be approximated according to a constant stress (Reuss model)[3] analysis as

$$G_{xz} = \frac{1}{1 - vf} \; G_M \tag{8}$$

where vf = reinforcement volume fraction
G_M = shear modulus of the matrix (psi)

The ratio of the beam deflection increments due to beam shear and beam moment are therefore (from eqs. 6, 7, and 8)

$$\frac{D_S}{D_M} = \frac{6}{5} (1 - vf) \left(\frac{t^2}{L^2}\right) \left(\frac{E3}{G_M}\right) \qquad (9)$$

This ratio may be evaluated approximately for vf = 0.40, t = .020 in., E3 = 20 x 10^6 psi, and G_M = 1.4 x 10^5 psi as

$$\frac{D_S}{D_M} = \frac{4}{L^2} \times 10^{-2} \qquad (9a)$$

Now for specimen span length L = 2.25 inches, this ratio is less than 1 percent and is therefore negligible. For specimen span length L = 0.75 inch, however, the ratio D_S/D_M is on the order of 10 percent. As a result, we may expect any samples tested in 3-point bending over a 0.75-inch span to exhibit a lower apparent stiffness. The predicted loss of bending stiffness of about 10 percent was, in fact, observed in specimen 121-72. In specimen 121-84, however, this reduction was much greater (i.e., E_F about 30 percent smaller than E3), and has not been explained.

The compressive strength of the laminates was significantly higher than the tensile strength for all the samples tested.

Fractography Studies

Fractography studies have shown that the mode of failure in compression was local geometric instability rather than decohesion or collapse of the material. It should be noted that the rather wide spread between highest and lowest test values (90.3 and 41.4 x 10^3 psi, respectively) reflected the local misalignment, nonplanarity, and/or departures from perfect symmetry of the reinforcement layers, rather than a wide variability of the material cohesive strength.

The short, unsupported compressive specimens failed typically by developing a local "kink" in mid-gage length (Figure 13, specimen 121-72F) or near one loaded edge (Figure 14, specimen 121-84D).

The 1.0-inch gage length compression specimens which were laterally supported by "fingers" (as shown in Figure 6b) typically failed by a double-kink confined to a small local region. This had the effect of extruding a small wedge, as shown in Figure 15 (specimen 121-72E), Figure 16 (specimen 121-84F), and Figure 17 (specimen 121-84G). In all cases, the loss of geometric stability could be identified as the immediate cause of failure.

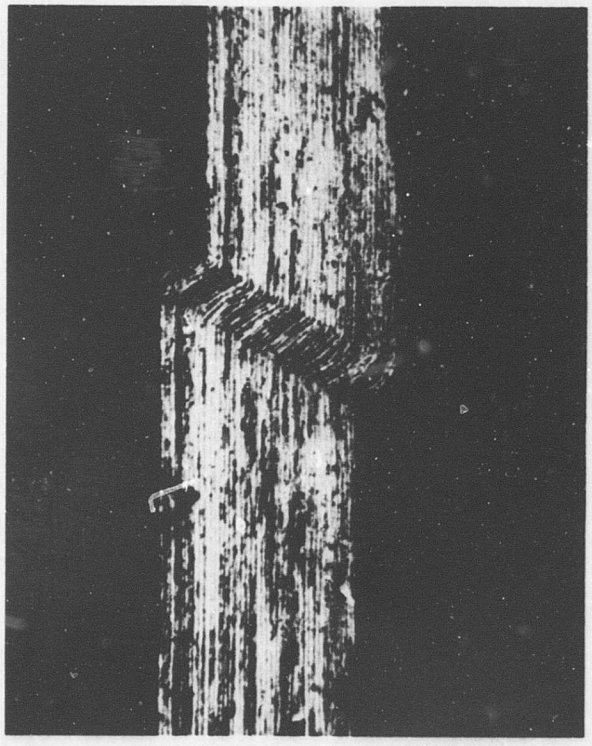


Figure 13. Compression Specimen 121-22F, Showing "Kink" in Mid-Gage Length. X 50.

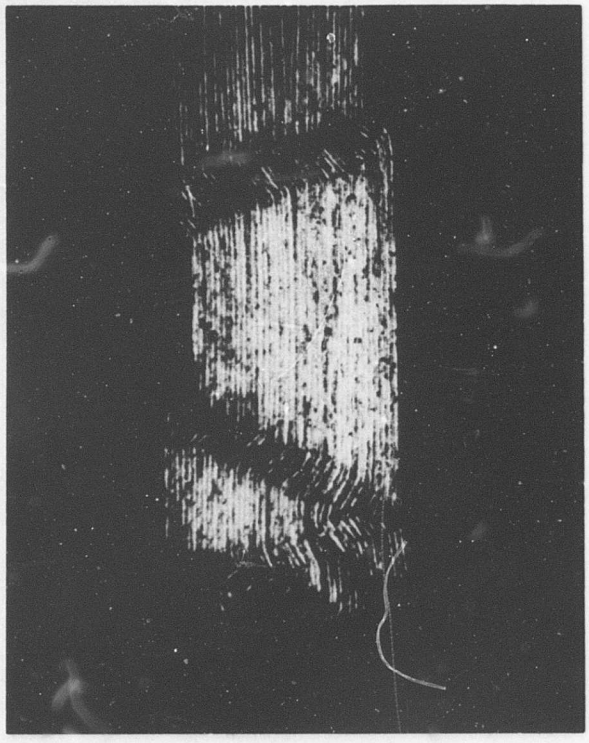


Figure 14. Compression Specimen 121-84D, Showing "Kink" Near One Loaded Edge. X 50.

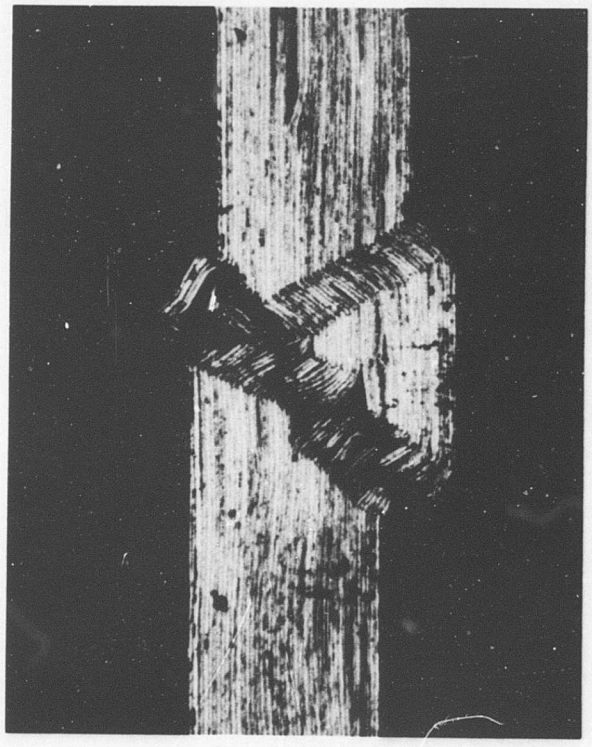


Figure 15. Compression Specimen 121-72E, Showing Extruded Wedge. X 50.

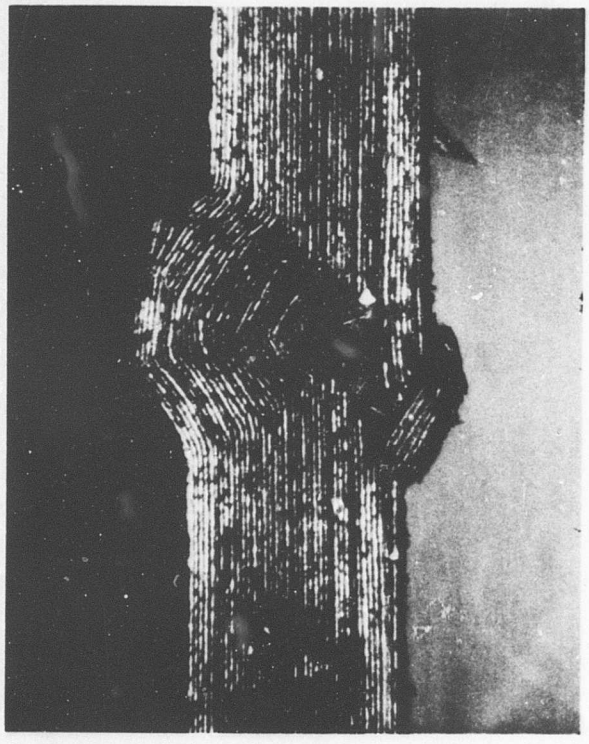


Figure 16. Compression Specimen 121-84F, Showing Extruded Wedge. X 50.

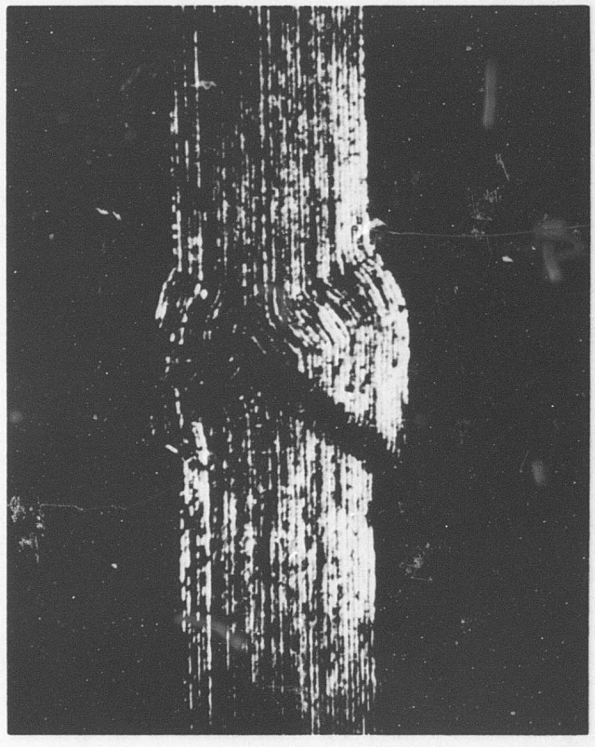


Figure 17. Compression Specimen 121-84G, Showing Extruded Wedge. X 50.

Sectioning of the interlaminar shear specimens showed that the fracture surface began as a longitudinal shear crack near the middle of the specimen, both tips of which then propagated as tension cracks outward toward the reentrant corners of the shear jig. It was interesting to note that the original shear crack did not follow any one boron/plastic interface but broke through the reinforcing layers instead. The mean crack plane was thus inclined about 7^0 to the plane of the laminations, as shown in Figure 18 for specimen 121-72h and in Figure 19 for specimen 121-84h. This effect, together with the measured shear strength values of about 12×10^3 psi, has indicated adequate adhesion at the interfaces and ample shear strength within the volume of the resinous matrix materials.

In the optical examination of the regions adjacent to the fracture zone of tensile specimens, two general techniques were used. The first involved direct examination of the fracture regions under the microscope together with polished sectioning of the tested specimen. While this type of examination yielded information regarding glue-line thickness, planarity of boron layers, and relative degrees of adhesion between the various layers in the composite, it was not particularly useful in indicating the causes for tensile fracture. A second procedure which was developed late in the program permitted a more direct observation of the internal structure of the laminate. It was found that if a drop of an aqueous hydrazine solution was applied to the area where delamination of the layers was initiated by inserting a sharp wedge, delamination could be made to continue along a single interface by maintaining a slight bending pressure. In this way, the internal interfaces of both fractured and untested laminates could be exposed for optical and microscopic examination. The most significant results of this approach are summarized as follows:

1. When boron-polyimide laminates which had been made up using the parallel lay-up techniques were delaminated, it was found that the degree of internal waviness was large. The maximum angle from the norm was as large as 5^0 (see Figures 20 and 21). On the other hand, when the cross-ply technique was used, this angle was much less. It would appear that the small curved surfaces generated by the copper screen carrier had a preferential direction for the main curvature. It is possible that this "fine scale" curvature was related to the larger scale of waviness in the laminate (Figure 21). It will be noticed that one of the surfaces is relatively flat, while the other surface is not flat in section (Figure 21). The curved surface

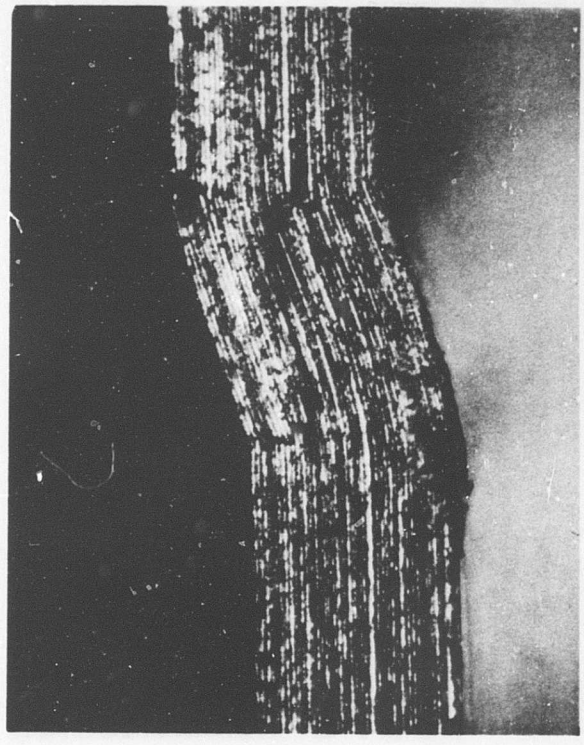


Figure 18. Interlaminar Shear Specimen 121-72H. X 50.

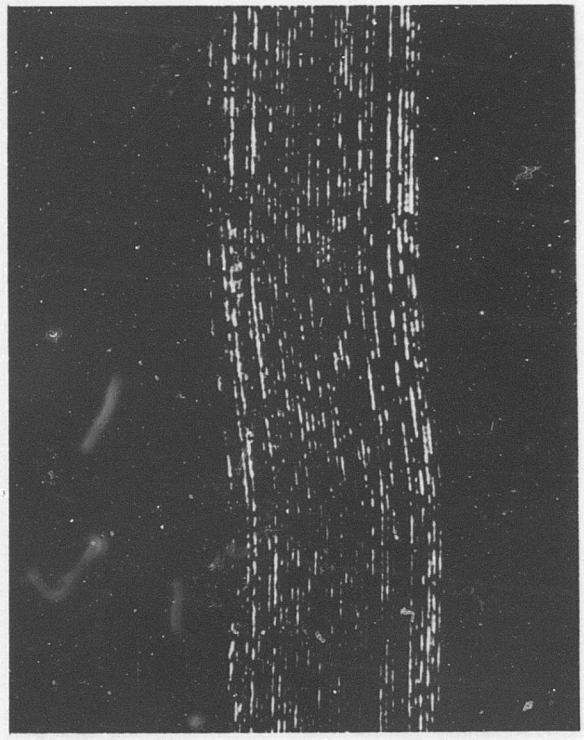


Figure 19. Interlaminar Shear Specimen 121-84H. X 50.

Figure 20. Section of Delaminated Test Specimen Showing Waviness of Internal Reinforcement Layers. X 22.

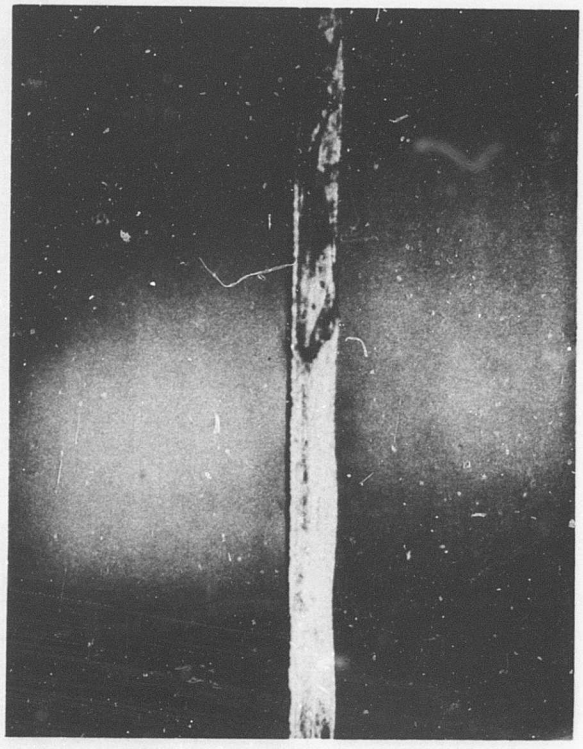


Figure 21. Section of Delaminated Test Specimen Showing Waviness of Internal Reinforcement Layers. X 20. Note right edge.

was an internal surface of the laminate, while the flat surface was an outer surface or platen surface of the laminate. These results indicated that external flatness of a laminate did not mean that all internal layers were parallel and planar. In addition, the curvature of the internal layer must have associated variations in glue-line thickness. It is likely that these types of imperfections would lead to decreases in both compressive and tensile properties.

2. The surfaces of the boron deposits facing the polyimide substrate were found to have long, fine-scale, parallel grooves (see Figure 22). These grooves were caused by draw marks in the polyimide substrate. The grooves did not appear to nucleate cracks. On the other hand, once cracks had been nucleated, they tended to propagate along these grooves (see Figure 23). From the results presented in Table VI, it is apparent that the tensile properties in the A direction - that is, parallel to the transport direction and parallel to the polyimide draw marks - are significantly higher than at right angles to this direction. It is possible that the draw marks are responsible for the difference in that they tend to give flakes with larger aspect ratios in the groove or A direction for parallel lay-up than in the B direction.

3. Optical examination of the surfaces of the internal layers showed that there were many folds and sharp creases in the internal reinforcement. These imperfections were undoubtedly smaller scale members of the larger wrinkles which had been largely eliminated from these laminates. Nevertheless, it was obvious that these folds often nucleated crack formation (see Figures 22 and 24).

4. The most significant result of the examination carried out using this delaminating procedure was that most of the cracks were nucleated by indentation (see Figures 25 and 26). It appeared that many of these indentations were caused by particulate material entrapped in the laminate. Two main sources of these particles are possible. First, the polyimide substrate has a high dielectric strength and electric resistivity. In handling, the surfaces develop and maintain high static charges. Consequently, it is possible that dust collected on the substrate both before placing the

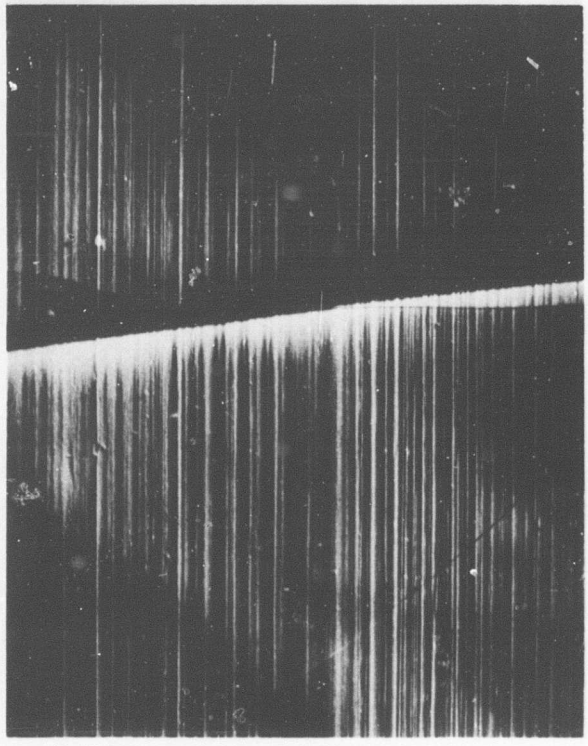


Figure 22. Draw Marks in Polyimide Film Reproduced by Boron Deposit. X 165.

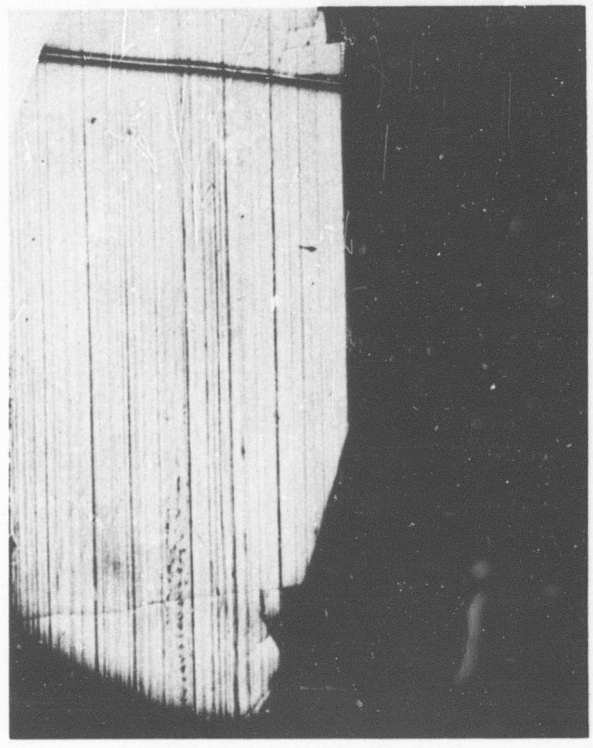


Figure 23. Propagation of Cracks Along Draw Mark Lines in Boron Film. X 80.

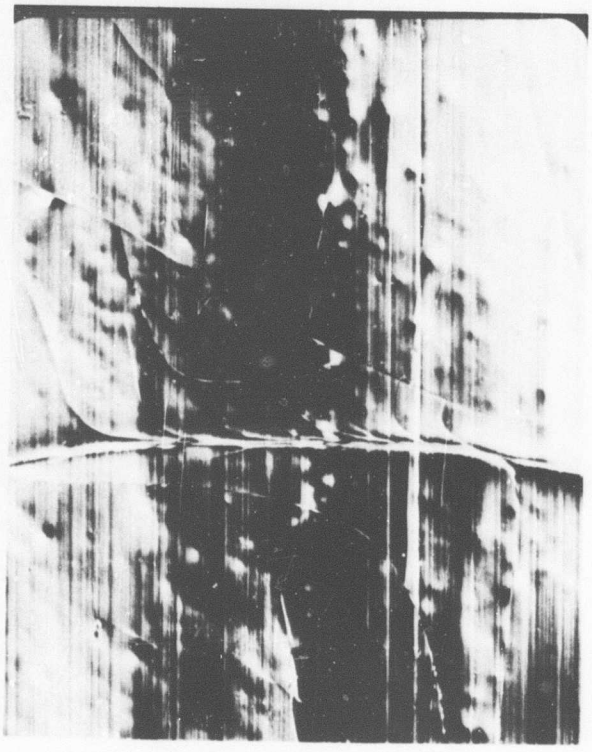


Figure 24. Fold and Crease Within Internal Reinforcement. X 165.

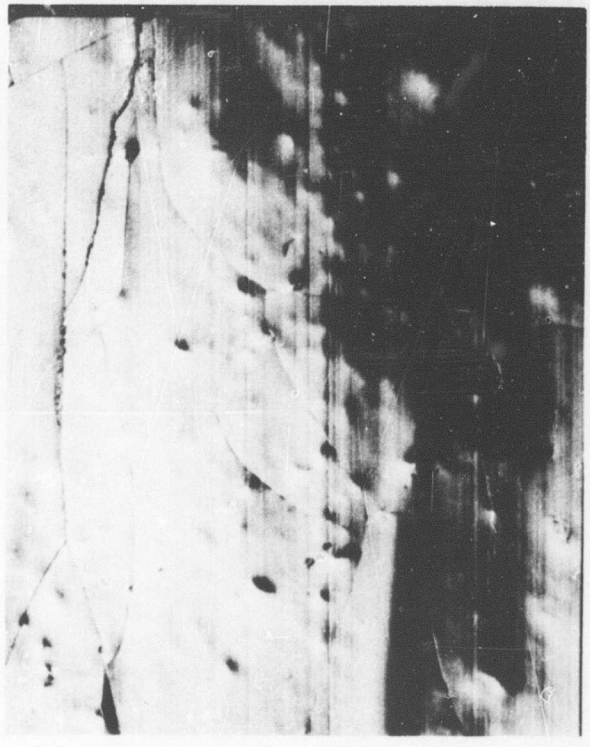


Figure 25. Nucleation of Cracks by Indentation. X 165.

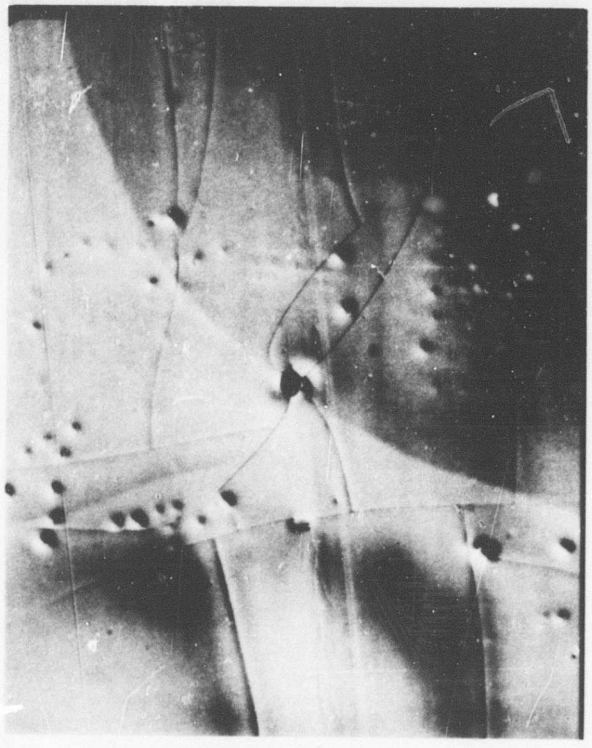


Figure 26. Nucleation of Cracks by Indentation. X 165.

substrate in the vacuum coater and before the film was coated. The other main possibility is dust which was incorporated in the laminate during laminate fabrication. It is apparent that rigorously clean conditions during coating and laminating should be maintained in future work. It is possible that higher tensile properties would result.

5. In the course of the work, a number of crack showers were observed (see Figure 27). The crack showers were generally arrested at a primary crack. This suggested that some degree of precracking of the sheets into large flakes may be beneficial for the purpose of crack arresting.

A comparison of the Section II results with those obtained with concept number 3 of Section I shows that the laminate with the highest tensile properties was laminate number 88-74, discussed in Section I. The stress-strain curve for this laminate is presented in Figure 28. The tensile strength was 56.6×10^3 psi and the modulus was 23.0×10^6 psi. As in most of the other laminates made from boron deposited on 1/4-mil polyimide film, the proportional limit strain was approximately 0.1 percent. On the other hand, laminate number 88-74 had a relatively high volume fraction of boron combined with a high failure strain. It was apparent that considerable differences in laminate tensile properties were associated with minor differences in the laminate making procedures. Although significant decreases in the magnitude of folds and wrinkles were achieved during this program, it was apparent that further improvements were required to achieve consistent results.

In order to compare the best laminate made under the present program with other planar isotropic materials of interest to the aircraft industry, Figure 29 has been prepared. This is a specific stress-strain curve for a common aluminum alloy, a common titanium alloy, a pseudo-isotropic (0 ± 60) boron filament composite, and the boron film composite. In terms of specific tensile strength, it is apparent that the best film laminate exceeds that of aluminum and is comparable to that of the boron filament composite. The best film composite is approximately 12 percent lower in specific strength than the titanium alloy. With regard to specific stiffness (the slope of the specific stress-strain curve), the boron film composite had the highest value. The film composite had a specific modulus over 3 times that of aluminum and titanium and approximately 2 times that of the boron filament composite.

Theoretically higher strengths are possible for the film reinforcement composite. It is possible that removal of the

Figure 27. Crack Shower. X 80.

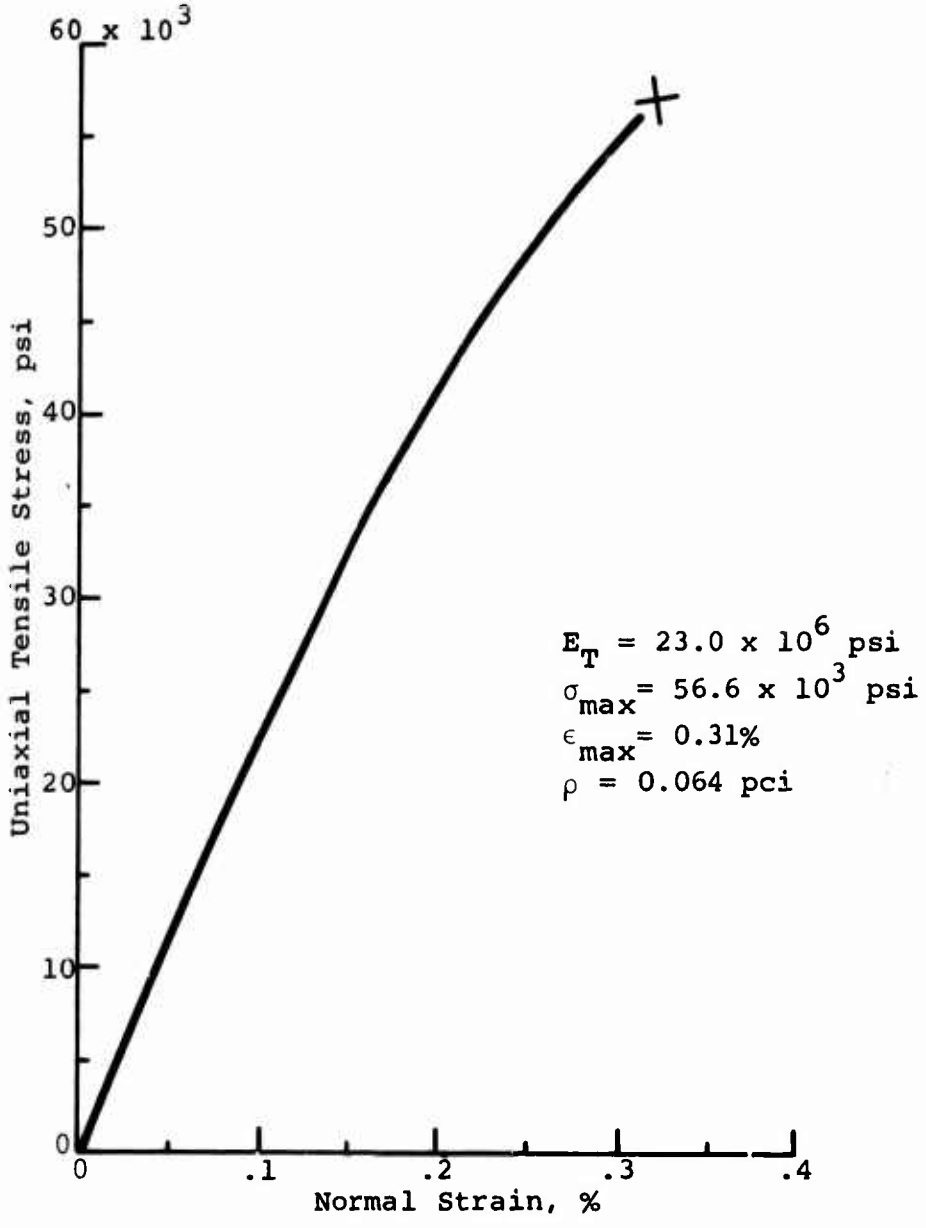


Figure 28. Stress-Strain Curve for Laminate 88-74.

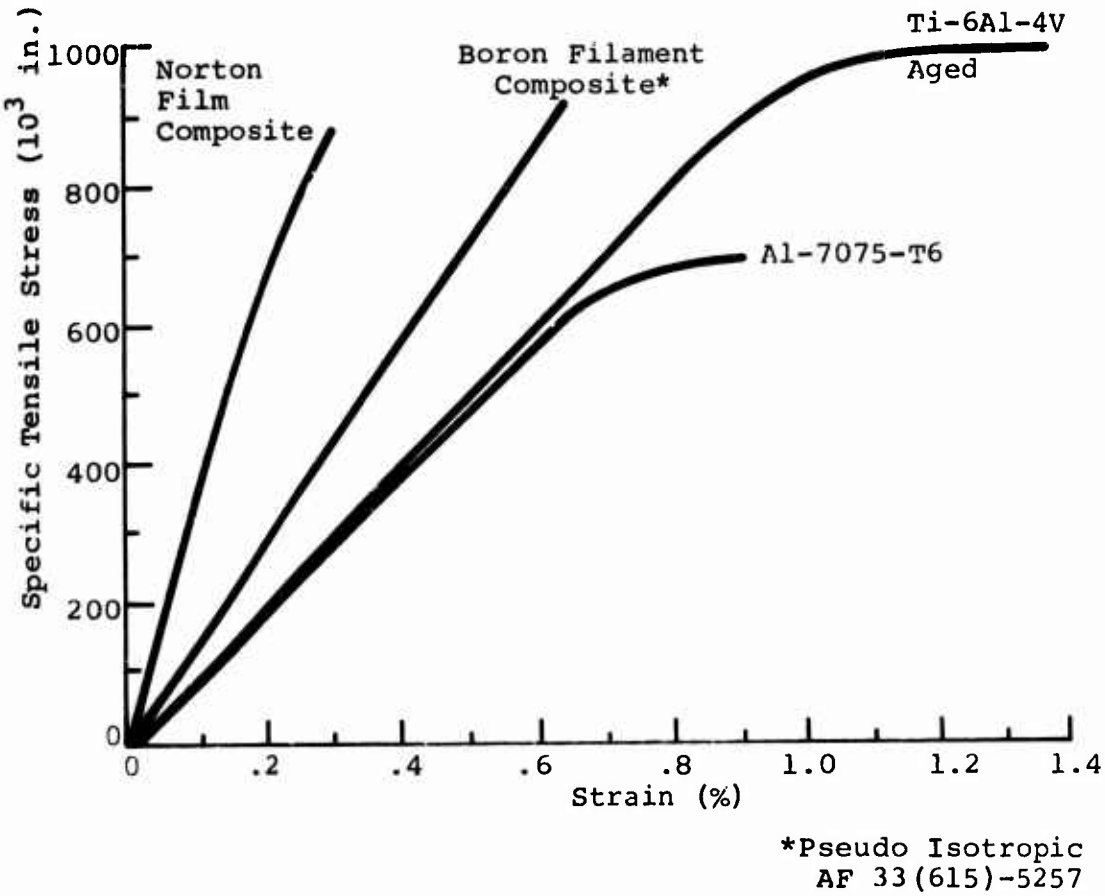


Figure 29. Comparison of Planar Isotropic Materials.

major flaws discussed earlier in the report would lead to higher tensile strengths.

SUMMARY OF RESULTS

The main results of the present program may be summarized as follows:

1. Boron films were vacuum evaporated onto two types of polyimide substrates. Most of the work employed a substrate which was 1/4 mil thick (DuPont-Kapton). A second substrate which received less attention but showed definite promise was a polyimide resin formed on a 2-mil aluminum substrate. However, a considerable degree of developmental work would have been required to produce experimental quantities of reinforcement with strength and modulus values equivalent to those obtained with the 1/4-mil substrate. Consequently, the latter part of the program concentrated on the 1/4-mil Kapton material.

2. Laminates were made by bonding together multiple layers of the boron-coated polyimide with epoxy resins. Laminates were then subjected to mechanical testing procedures. The maximum results obtained during the program were as follows:

   a. Tensile strength - 56.6×10^3 psi

   b. Tensile modulus - 23.0×10^6 psi

   c. Flexural strength - 57.4×10^3 psi

   d. Flexural modulus - 23.1×10^6 psi

   e. Compressive strength - 90×10^3 psi

   f. Compressive modulus - 24.5×10^6 psi

   g. Shear modulus - 7.7×10^6 psi

   h. Interlaminar shear strength - 13.8×10^3 psi

   The density of the laminates with the above properties was approximately 0.064 pci.

3. The laminates showed a high degree of isotropy in the plane of the laminates. However, the tensile strengths of the laminates were lower (approximately 30%) when tested at right angles to the direction of transport of the substrate through the vacuum coating chamber. Although the cause for this variation was not

determined, it is likely that it is associated with the longitudinal draw marks in the 1/4-mil polyimide substrate.

4. Vacuum coating and laminating procedures were developed to increase the flatness of the individual layers within the laminate and to decrease the variations in the thickness of the bonding adhesive.

5. Optical examination of the laminate test specimens indicated that laminate tensile and compressive strengths were adversely affected by:

   a. Folds and waviness of the reinforcement layers within the laminate.

   b. Draw marks on the polyimide substrate.

   c. Dust and foreign inclusions within the laminate.

RECOMMENDATIONS

It is recommended that the following approaches for increasing the strength of film reinforcement laminates receive experimental investigation:

1. Maintain dust-free conditions during both the coating and laminating operations to eliminate the entrapping of particulate matter between layers of reinforcement.

2. Form a flat tape made of 2 or 3 layers of boron-coated polyimide film. Subsequent lamination of multiple layers of tape should produce laminates with higher degrees of internal planarity.

3. Eliminate the draw marks on the 1/4-mil polyimide substrate.

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13. ABSTRACT

The main objective of this program was to develop improved mechanical properties of boron thin-film reinforcement. Optical examinations of laminates made by bonding together multiple layers of vacuum evaporated boron supported on polyimide substrates in earlier programs had shown significant variations in glue-line thickness and reinforcement planarity. The present program emphasized methods of reducing these types of laminate imperfections. Three approaches were experimentally tested. The best results were obtained with laminates made with polyimide film (Kapton), 1/4 mil thick, as the substrate. This was vacuum coated on both sides with boron using a screen carrier in the vacuum coating operation. Multiple layers were bonded together to make laminates for mechanical testing. The optimum mechanical properties obtained were as follows: tensile strength, 56.6 x 10^3 psi; tensile modulus, 23.0 x 10^6 psi; flexural strength, 57.4 x 10^3 psi; flexural modulus, 23.1 x 10^6 psi; compressive strength, 90 x 10^3 psi; compressive modulus 24.5 x 10^6 psi; shear modulus, 7.7 x 10^6 psi; and interlaminar shear strength, 13.8 x 10^3 psi. The density of a typical laminate was 0.064 pci.

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
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Thin-film						
Vacuum evaporation						
Laminate						
Polyimide						
Mechanical properties						
Strength						
Modulus						
Epoxy						
Isotropic						